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ENGINEERING AND DEVELOPMENT SUPPORT OF GENERAL DECON TECHNOLOGY FOR THE DARCOM INSTALLATION RESTORATION PROGRAM

Task 10. Analysis of LAAP Lagoon Water

Lagoon #9

Circulation Copy

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Submitted to:

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Task 10 - Analysis of LAAP Lagoon Water

A sample of lagoon water was received on 7 May 1981 from Louisiana AAP. This sample was immediately refrigerated until analysis.

The lagoon water sample was a deep red-orange color and contained no particulates. The pH of the sample as received was 8.0. The following analyses were run on this sample:

- COD
- lead and zinc
- neutral, basic and acidic ethyl ether exractions followed by GC-MS of the concentrated extracts to identify components
- derivatization of the acidic extract and GC/MS of the derivative to identify acidic components
- quantitation of RDX, TNT, and 1,3,5-trinitrobenzene (TNB)

A. Experimental Proceedure

The lead and zinc concentrations in the lagoon water were determined by atomic absorption according to approved USATHAMA method #4A. Detection limits for lead and zinc by this method are 0.250 mg/L and 0.177 mg/L, respectively. The COD contents of the lagoon water was determined according to the procedure set forth in Standard Method No. 508.

The netural extraction was performed by placing 10 ml of the lagoon water in a 20 ml culture vial with screw cap and teflon liner. The water was extracted with 1 ml of ethyl ether (A.C.S. reagent, Fisher Scientific). The ether layer was drawn off and placed in a Kuderna-Danish evaporator. The water sample was re-extracted with a second ml of ether and the extracts combined. The combined extracts were evaporated to 0.1 ml (100:1 concentration) and injected into a Hewlett-Packard 5992A GC-MS with a 9825A calculator and 9866B printer. The following GC-MS conditions were used:

Column:	2% Dexsil 300 GC on 90/100 mesh Anakrom Q in a 2 mm ID x 1/2 in OD x 6 ft long column
temperature:	injection port - 210°C oven - programmed from 140 to 260°C at 15°C/min

For the basic extraction, 10 ml of lagoon water were placed in a culture vial with screw cap and teflon liner. Sodium hydroxide (5 N) was added to the 10 ml to a pH of 11.0. The resulting solution was extracted, concentrated, and chromatographed as described above for the neutral extraction.

The acidic extraction was performed in the same manner as the basic extraction except that hydrochloric acid (6N) was added to the solution until a pH of 2 was reached. The resulting solution was extracted and chromatographed as described above. A second acid extract of 10 ml of lagoon water was performed. This extract was subjected to esterification using the BF_3-methanol microesterification reagent (Supelco, Inc.). The ether extract was evaporated to dryness and the solids dissolved in 2 ml of benzene. This solution was added to the reagent, and boiled for 3 minutes. One ml of water was then added to stop the reaction. The layers were separated and the benzene layer concentrated before injecting into the GC/MS.

Identification of the components of the lagoon waters was accomplished by comparing the mass spectrum of the water components with that of the actual compound, if it was available. If the compound was not available, identification was made through comparison of the spectrum with the EPA-NIH files. For those compounds not in the EPA-NIH files, tentative identification was made through scientific evaluation of the mass spectra.

RDX, TNT, and TNB in the lagoon water were quantitated using HPLC. A reverse phase C-18 water radial compression column was used on a Perkin-Elmer Model #601 HPLC with a LC-55 variable wavelength detector and Cole-Palmer strip chart recorder. The carrier was 50% methanol water at a flow rate of 1.5 mL/min. UV detection was accomplished at 230 nm

B. Results

The lead, zinc, COD, RDX and TNT levels in the lagoon water were:

- lead < 0.250 mg/L
- zinc < 0.177 mg/L
- COD 42,336 mg/L
- RDX 89.5 mg/L
- TNT 26.3 mg/L
- TNB 12.0 mg/L

The chromatograms and the mass spectra of the GC peaks of the four extraction solutions are presented in Figures 1-4.

The neutral extract (Figure 1) had three major peaks (spectra #13, 14-15 and 17). Spectra #14, 15 and 17 were identified as TNT, 1,3,5-trinitrobenzene and RDX, respectively, by comparison with authentic SARMS of these compounds (see Figures 5-10). TNT and 1,3,5-trinitrobenzene are not well separated on this column. Both are present in the 12-27 ppm range in the lagoon water. Spectra #13 was tentatively identified as 2,6-bis(1,1-dimethylethyl)-4-methyl phenol by comparison with the EPA-NIH published spectra. It is expected that this is not the exact compound, but alkyl substituted phenol is highly indicated. The mass spectra of many of the alkyl substituted phenols are very similar. In addition to the major peak, a small peak (#18) was identified as 2-amino-4,6-dinitrotoluene by comparison with an authentic standard of the material.

The base extract (Figure 2) showed the presence of 2 main peaks: 1,3,5-trinitrobenzene and RDX. The 2-amino-4,6-dinitrotoluene and TNT were also present in this extract as well as small amounts of hydrocarbons and a compound that is similar to tributyltin chloride (spectra #47).

The acidic extract had numerous components as shown in Figure 3. The spectrum numbers and identification are listed below:

33 probably an isomer of di-t-butylmethyl phenol (tentative)
34 dinitrobenzene
35 C_{16} hydrocarbon
36 chlorodinitrobenzene
37 C_{17} hydrocarbon
38 dibutyltin chloride (tentative)
39 1,3,5-trinitrobenzene
40 unknown
41 phthalate ester
42 RDX
43 unknown

The derivatized acid extract had many of the components found in the underivatized acid extract plus several methyl esters of organic acids. The spectrum numbers and identification are listed below:

2 chlorodinitrobenzene
3 unknown? too small to get a good spectrum
4 a methyl ester of an organic acid - probably myristic acid
5 TNT
6 methyl palmitate - acid palmitate
7 RDX
8 unknown
9 methyl stearate - acid stearic acid

In summary, the lagoon water contains a wide variety of components. The major components are TNT, TNB and RDX. These components are present in the 12-90 mg/L range.

FIGURE 2 - NEUTRAL EXTRACT OF LABP LAGOON WATER

Next Spectrum recorded will be 13 FIGURE 1

** CONDITIONS FOR RUN # 1 dated: 5/26/1981 [illegible]

TEMP1 Deg.	TIME1 min.	RATE Deg/min.	TEMP2 Deg.	TIME2 Min.	INJ.PORT Deg.	MS.OVEN Deg.	SOLVENT Min.	RUN TIME Min.
140	0.0	15.0	260	32.0	210	280	0.0	30.0

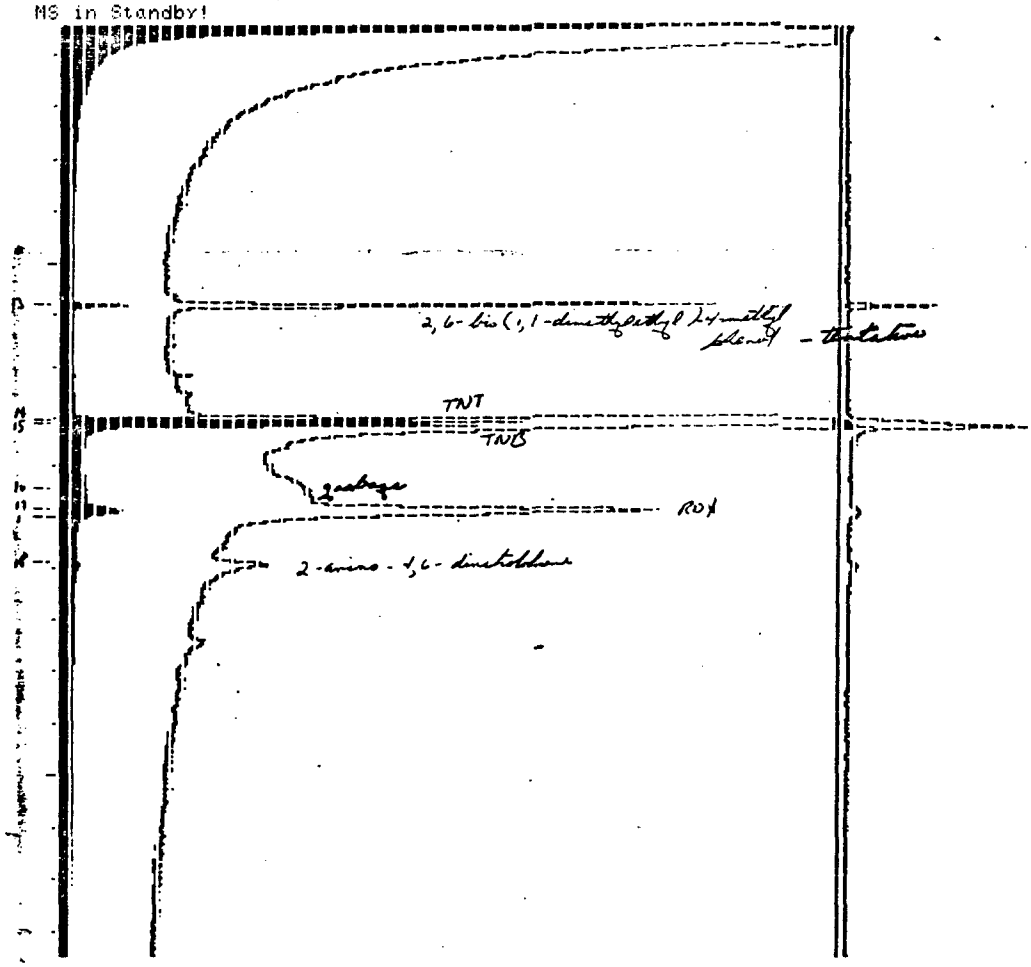
MS PEAK DETECT THRESHOLD = 5.0 linear counts
FLOW RATE = 16 ml/min
SAMPLES PER .1 AMU = 8 SCAN SPEED = 200 amu/sec
ELECTRON MULTIPLIER = 2000 volts
GC PEAK DETECT THRESHOLD = 400 TRIGGERED ON TOTAL ABUNDANCE
REAL TIME STRIPPING OF VALLEYS FROM PEAKS

SAMPLE NAME neutral ether extract of LABP lagoon water

OPERATOR RJ

TOTAL ABUNDANCE FROM 40 TO 460 amu
Full Scale= 9920
ION 121.0
Full Scale= 150

Excess Source Pressure!
MS in Standby!
No Emission Current!
Detector Problem!
MS in Standby!
Excess Source Pressure!
No Emission Current!
MS in Standby!
Excess Source Pressure!
No Emission Current!
MS in Standby!



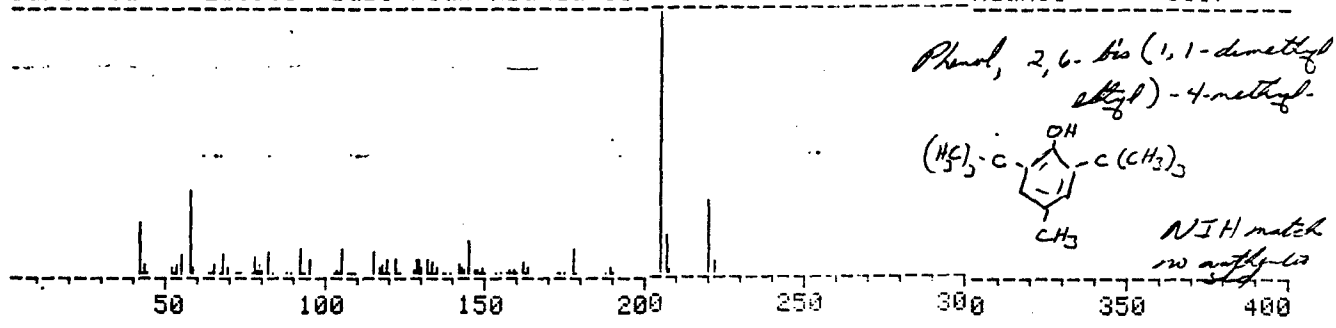
SPECTRA SAVED: Run # 1

FIGURE 2 (cont'd)

Spectrum	Ret. Time	Total Abund.	Relative Abund.	Base Peak	
13	5.8	5187	13.8%	205.05	
14	8.0	37490	100.0%	209.95	- TNT
15	8.1	17727	47.3%	74.95	- TNB
16	9.4	430	1.1%	42.00	
17	9.8	4352	11.6%	41.90	- RDX
18	10.8	534	1.4%	77.95	- 2-amino-4,6-dinitrotoluene

Spectra Plot/Tab Program: [Rev 8/4/78]

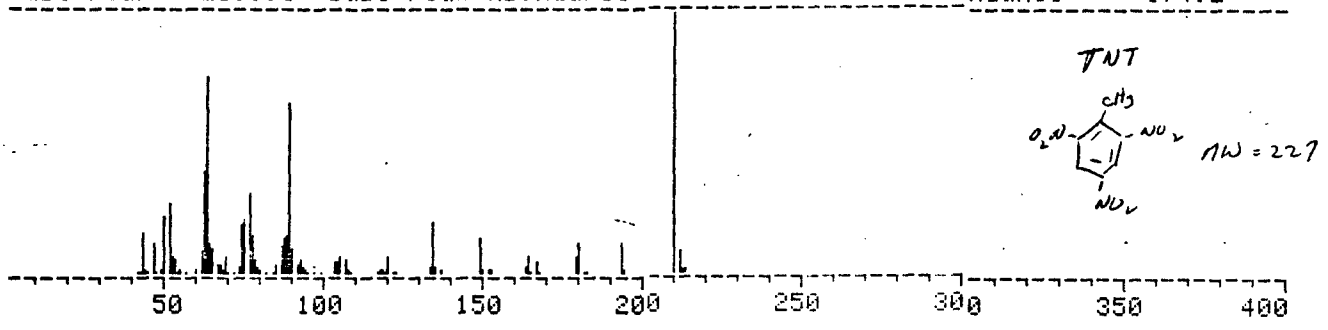
** Spectrum # 13 ** Sample # 1 Retention Time = 5.8 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 135
File type = linear
Base Peak = 205.05 Base Peak Abundance = 1152 Total Abundance = 5187



Phenol, 2,6-bis(1,1-dimethylethyl)-4-methyl-

NIH match no methyls

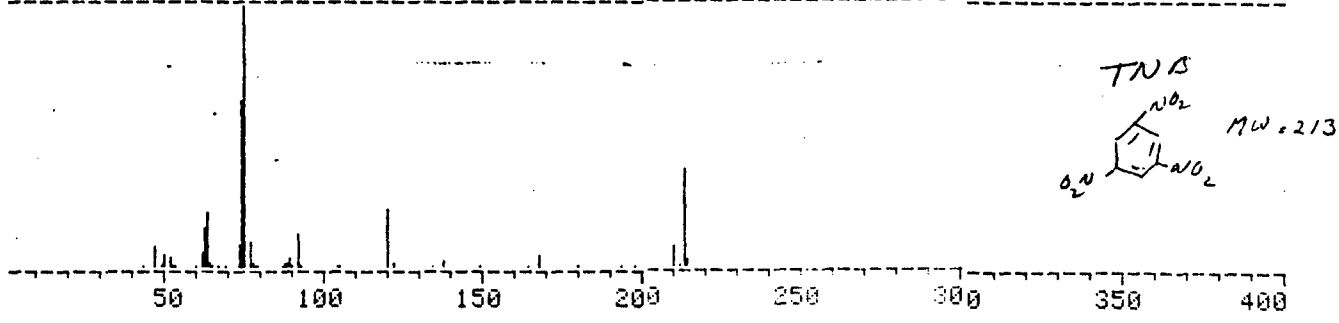
** Spectrum # 14 ** Sample # 1 Retention Time = 8.0 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 147
File type = linear
Base Peak = 209.95 Base Peak Abundance = 4912 Total Abundance = 37492



TNT

MW = 227

** Spectrum # 15 ** Sample # 1 Retention Time = 8.1 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 122
File type = linear
Base Peak = 74.95 Base Peak Abundance = 4712 Total Abundance = 17727

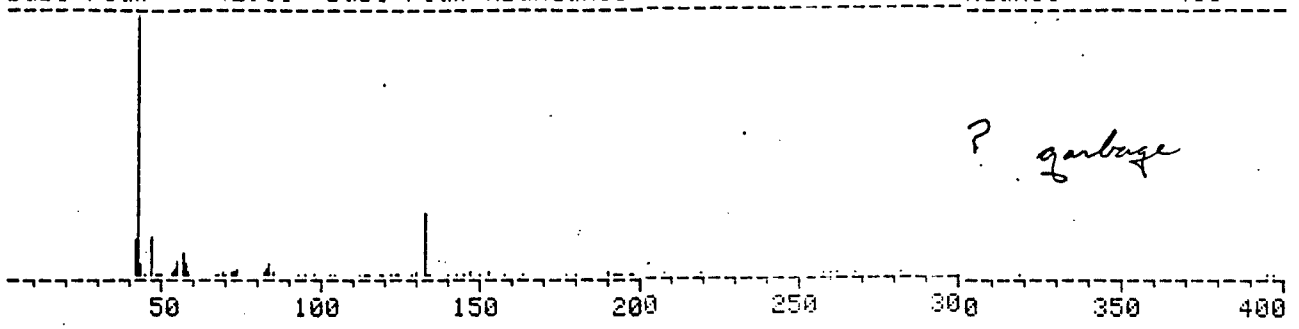


TNB

MW = 213

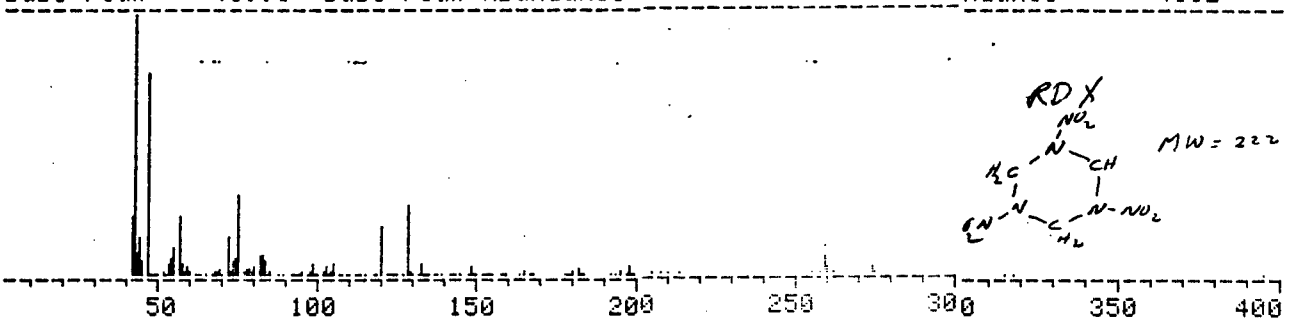
FIGURE 3 (cont.)

** Spectrum # 16 ** Sample # 1 Retention [illegible] minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 71
File type = linear
Base Peak = 42.00 Base Peak Abundance = 176 Total Abundance = 430



? garbage

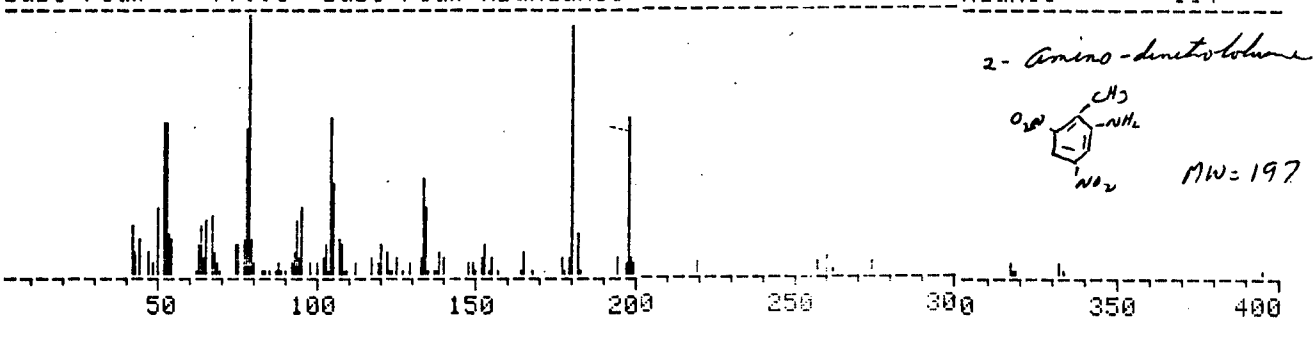
** Spectrum # 17 ** Sample # 1 Retention Time = 9.8 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 157
File type = linear
Base Peak = 41.90 Base Peak Abundance = 748 Total Abundance = 4352



RDX

MW = 222

** Spectrum # 18 ** Sample # 1 Retention Time = 10.8 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 100
File type = linear
Base Peak = 77.95 Base Peak Abundance = 43 Total Abundance = 534



2- Amino-dinitrotoluene

MW = 197

SPECTRA SAVED: Run # 1

FIGURE 2 - BASE EXTRACT OF LAAP LAGOON WATER

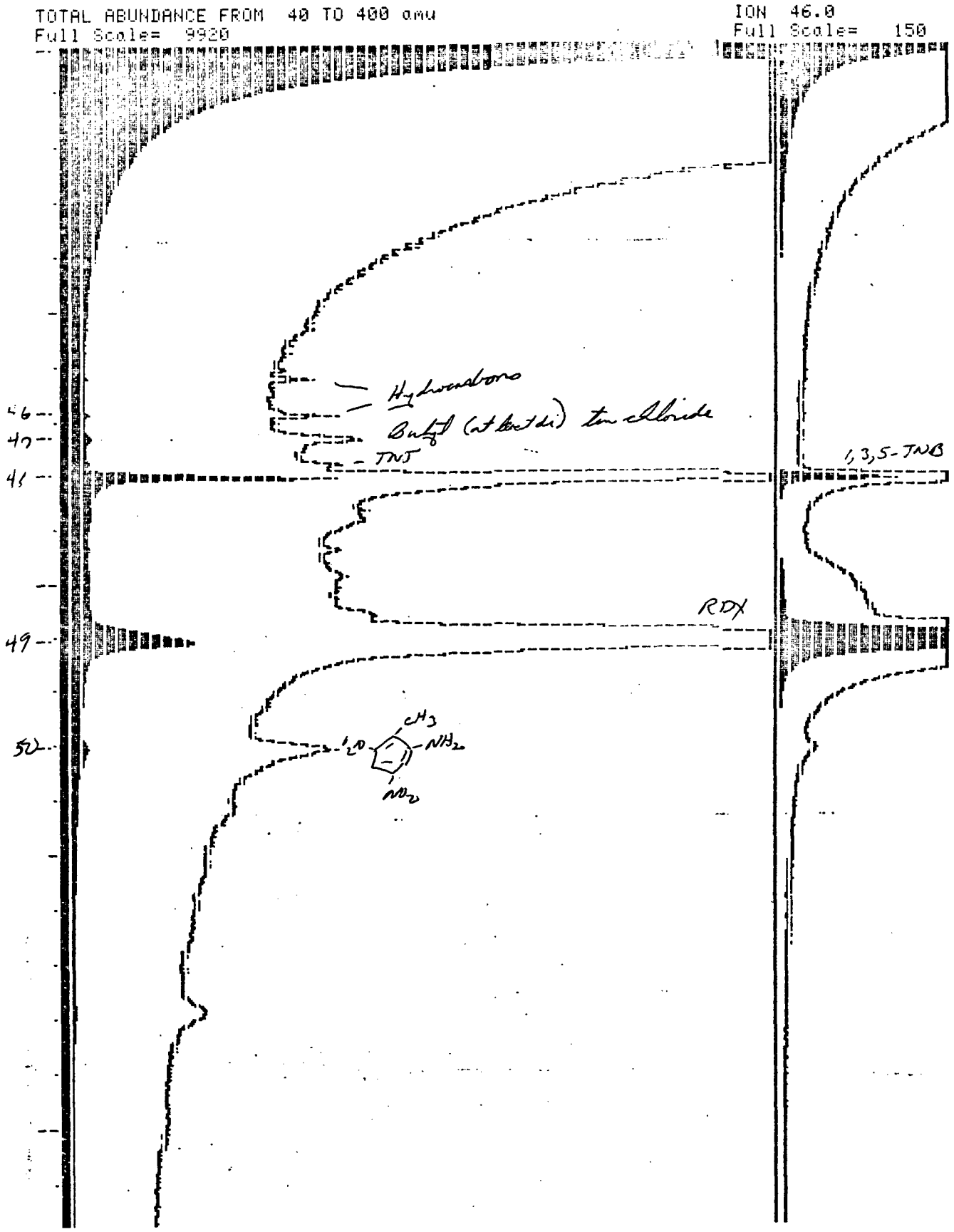
** CONDITIONS FOR RUN # 1 dated: 5/26/1981 Tuesday

TEMP1 Deg.	TIME1 min.	RATE Deg/min.	TEMP2 Deg.	TIME2 Min.	INJ.PORT Deg.	MAN.OVEN Deg.	SOLVENT Min.	RUN TIME Min.
160	1.0	15.0	240	32.0	210	290	0.0	30.0

MS PEAK DETECT THRESHOLD = 5.0 linear counts
FLOW RATE = 16 ml/min
SAMPLES PER .1 AMU = 8 SCAN SPEED = 2000 amu/sec
ELECTRON MULTIPLIER = 2000 volts
GC PEAK DETECT THRESHOLD = 800 TRIGGERED ON TOTAL ABUNDANCE
REAL TIME STRIPPING OF VALLEYS FROM PEAKS

SAMPLE NAME Figure 2

OPERATOR BASE EXTRACT (reconstituted)



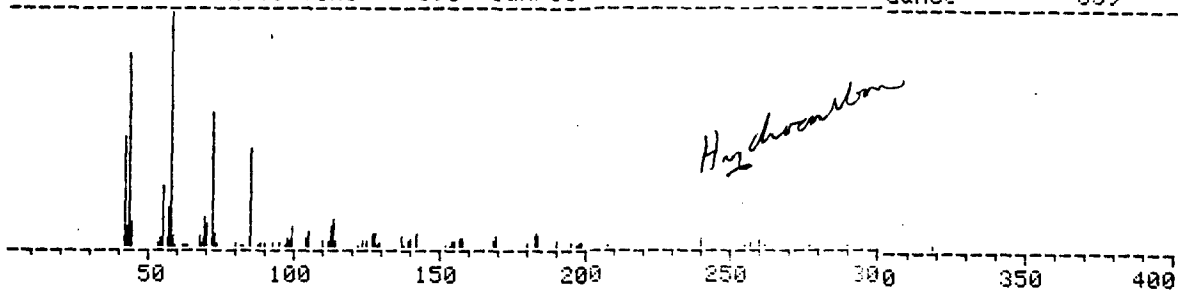
SPECTRA SAVED: Run # 1

FIGURE 2 CONT.

Spectrum	Ret. Time	Total Abund.	Relative Abund.	Base Peak
45	0.1	3078	13.3%	48.85
46	6.8	659	2.9%	56.95
47	7.3	952	4.1%	40.95
48	7.9	23076	100.0%	74.90
49	11.0	8449	36.6%	41.95
50	13.0	840	3.6%	179.95

*** LIBRARY SEARCH [rev. 1/1/78]

Spectrum # 46 Ret. Time= 6.8 Sample # 1 Total Abundance = 659



Hydrocarbon

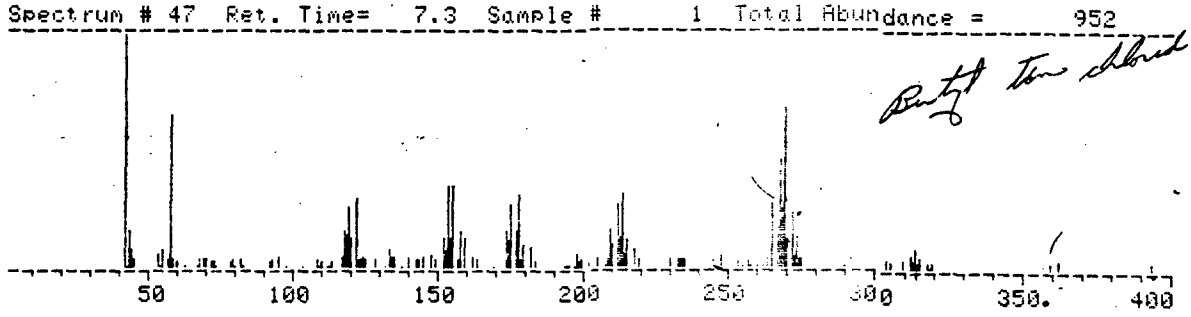
10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
41.0	51	47.2	34.0
43.0	89	82.4	62.2
55.0	28	25.9	25.0
56.0	18	16.7	16.4
57.0	108	100.0	100.0
71.0	62	57.4	71.5
85.0	45	41.7	62.1
112.0	10	9.3	18.2
113.0	13	12.0	23.9
182.0	7	6.5	20.7

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
5	0.9902	226.0
7	0.9875	254.0
3	0.9857	198.0
4	0.9854	212.0
6	0.9790	240.0
8	0.9766	268.0
1	0.9704	170.0
438	0.9569	312.0
436	0.9524	256.0
437	0.9493	284.0

Spectrum # 47 Ret. Time= 7.3 Sample # 1 Total Abundance = 952



Butyl Tin chloride

Figure 2 cont'd

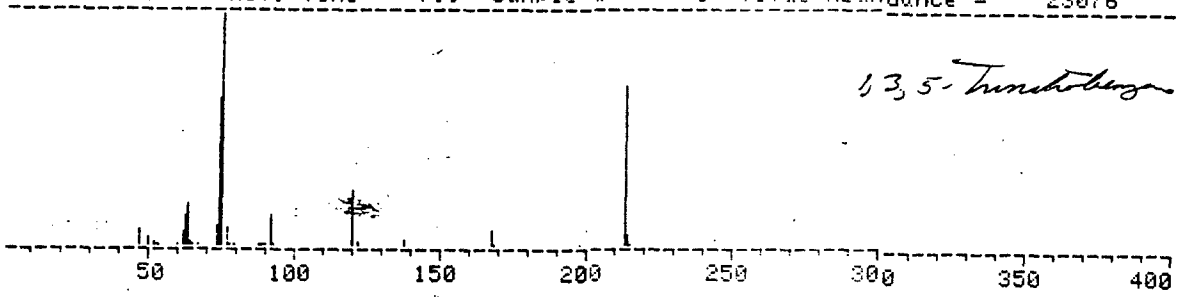
10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
153.0	27	51.9	29.5
155.0	27	51.9	29.9
177.0	24	46.2	30.4
211.0	21	40.4	31.7
213.0	24	46.2	36.5
265.0	21	40.4	39.8
267.0	37	71.2	70.6
268.0	19	36.5	36.4
269.0	52	100.0	100.0
271.0	18	34.6	34.9

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
280	0.2189	278.0
469	0.2072	153.0
349	0.2067	154.0
372	0.2003	186.0
484	0.1947	220.0
277	0.1708	225.0
109	0.1549	320.0
513	0.1534	266.0
403	0.1517	296.0
134	0.1468	192.0

Spectrum # 48 Ret. Time= 7.9 Sample # 1 Total Abundance = 23076



10 peaks used for search:

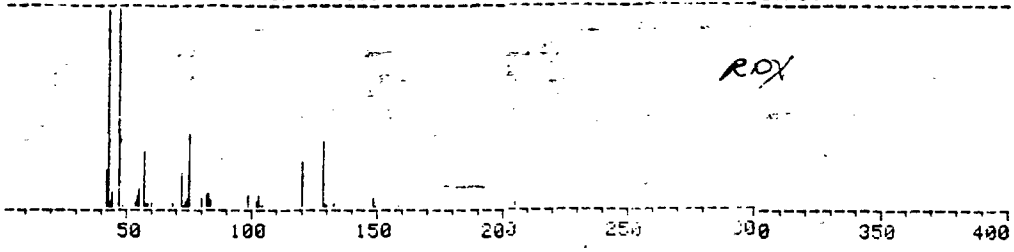
Mass	Linear Abund	% Abund	Significance
62.0	818	13.4	5.8
63.0	1092	17.9	7.8
74.0	3877	63.5	32.6
75.0	6109	100.0	52.0
76.0	507	8.3	4.4
91.0	772	12.6	8.0
120.0	1437	23.5	19.6
167.0	423	6.9	8.0
213.0	4136	67.7	100.0
214.0	313	5.1	7.6

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
202	0.6552	260.0
493	0.4781	180.0
450	0.4698	312.0
447	0.4442	256.0
451	0.4318	326.0
286	0.4313	298.0
448	0.4300	284.0
449	0.4243	298.0
463	0.4237	438.0
284	0.4195	242.0

FIGURE 2 (cont.)

Spectrum # 49 Ret. Time= 11.0 Sample # 1 Total Abundance = 8449



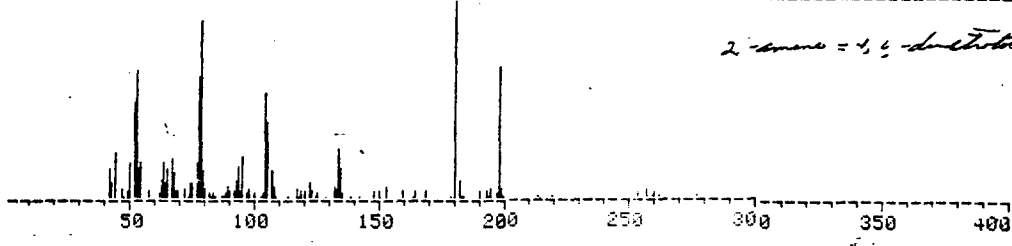
10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
41.0	335	18.5	16.7
42.0	1809	100.0	92.1
46.0	1793	99.1	100.0
56.0	509	28.1	34.6
71.0	326	18.0	28.1
75.0	678	37.5	61.7
82.0	132	7.3	13.1
120.0	409	22.6	59.5
128.0	602	33.3	93.4
148.0	95	5.3	17.0

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
133	0.2705	164.0
467	0.2495	138.0
202	0.2223	260.0
348	0.2013	128.0
135	0.1876	145.0
106	0.1277	177.0
341	0.1263	101.0
268	0.1239	138.0
298	0.1189	108.0
215	0.1147	136.0

Spectrum # 50 Ret. Time= 13.0 Sample # 1 Total Abundance = 840



10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
51.0	38	47.5	13.5
52.0	51	63.8	18.4
77.0	49	61.3	26.2
78.0	71	88.8	38.5
95.0	17	21.3	11.2
104.0	42	52.5	30.3
105.0	30	37.5	21.9
133.0	20	25.0	18.5
180.0	80	100.0	100.0
197.0	53	66.3	72.5

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
218	0.4744	162.0
490	0.4312	106.0
469	0.4283	153.0
430	0.4239	182.0
494	0.4205	140.0
470	0.4129	120.0
227	0.3981	148.0
322	0.3814	356.0
495	0.3756	210.0
432	0.3754	123.0

FIGURE 3. ACID EXTRACT OF IAAP LAGOON WATER

** CONDITIONS FOR RUN # 1 dated: 5/15/1981 Friday

TEMP1 Deg.	TIME1 min.	RATE Deg/min.	TEMP2 Deg.	TIME2 Min.	INJ.PORT Deg.	MAN.OVEN Deg.	SOLVENT Min.	RUN TIME Min.
160	1.0	15.0	240	32.0	210	280	0.0	30.0

MS PEAK DETECT THRESHOLD = 5.0 linear counts
FLOW RATE = 16 ml/min
SAMPLES PER .1 AMU = 8 SCAN SPEED = 200 amu/sec
ELECTRON MULTIPLIER = 1800 volts
GC PEAK DETECT THRESHOLD = 800 TRIGGERED ON TOTAL ABUNDANCE
REAL TIME STRIPPING OF VALLEYS FROM PEAKS

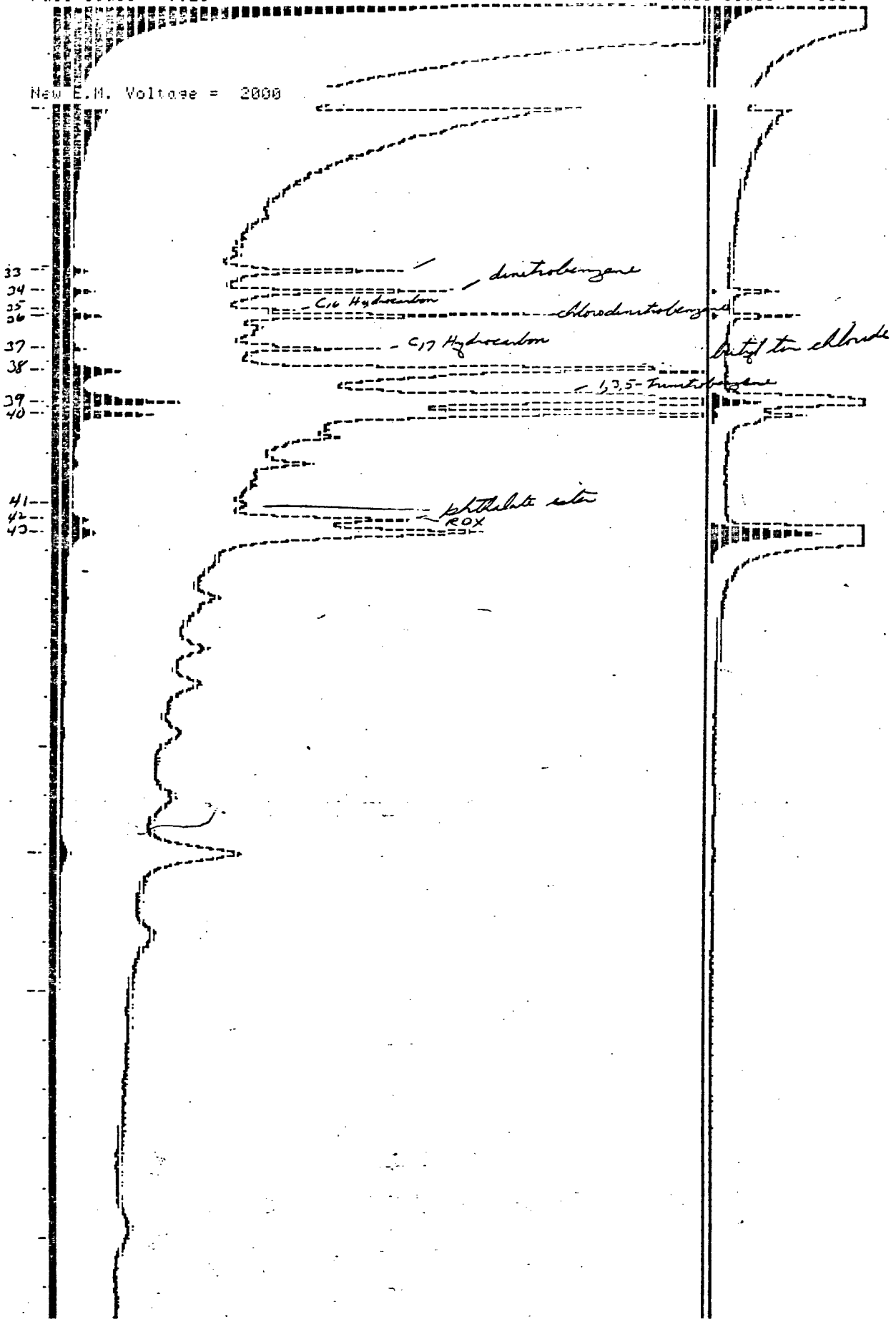
SAMPLE NAME IAAP Lagoon Water Acid Extract

OPERATOR BJ

TOTAL ABUNDANCE FROM 40 TO 400 amu
Full Scale= 9920

ION 46.0
Full Scale= 150

New E.M. Voltage = 2000



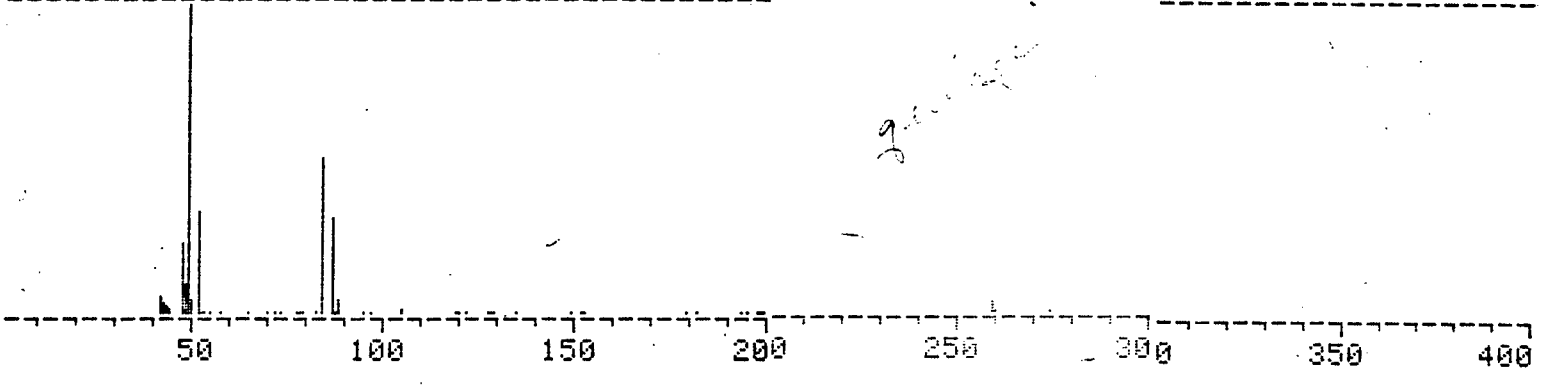
SPECTRA SAVED: Run # 1

FIGURE 3 (cont'd)

Spectrum	Ret. Time	Total Abund.	Relative Abund.	Base Peak
32	1.8	2159	21.2%	48.85
33	5.0	1937	19.0%	40.95
34	5.4	2184	21.4%	49.95
35	5.8	624	6.1%	56.95
36	6.0	4792	46.9%	74.90
37	6.7	1295	12.7%	56.95
38	7.1	5238	51.3%	40.95
39	7.8	10207	100.0%	74.90
40	8.1	7031	68.9%	183.95
41	10.3	2040	20.0%	149.00
42	10.6	1729	16.9%	45.95
43	17.1	851	8.3%	56.95

*** LIBRARY SEARCH [rev. 1/1/78]

Spectrum # 32 Ret. Time= 1.8 Sample # 1 Total Abundance = 2159



10 peaks used for search:

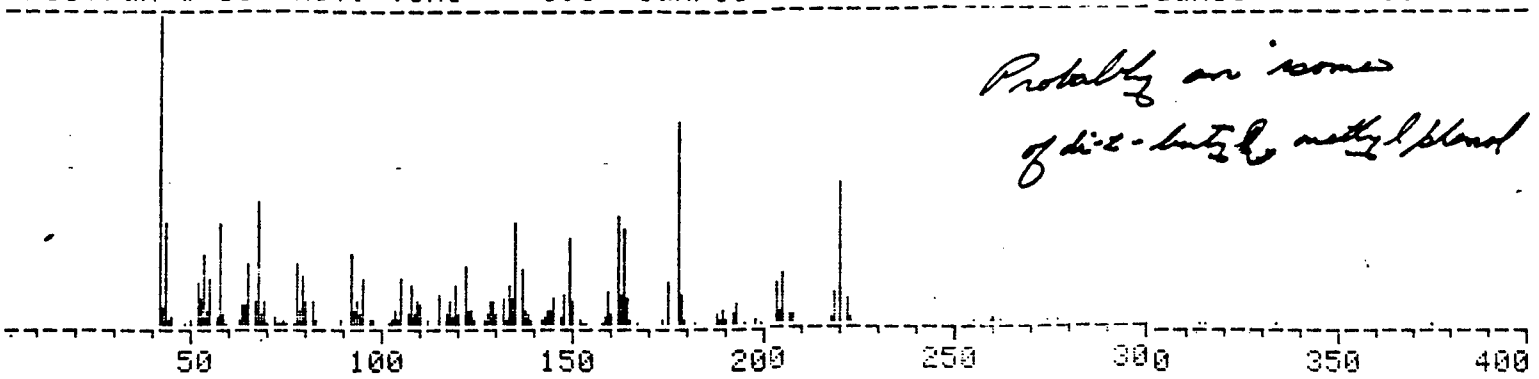
Mass	Linear Abund	% Abund	Significance
41.0	43	6.4	5.4
47.0	151	22.5	21.6
48.0	69	10.3	10.1
49.0	670	100.0	100.0
51.0	222	33.1	34.5
84.0	337	50.3	86.2
86.0	210	31.3	55.0
88.0	33	4.9	8.8
259.0	30	4.5	23.7
274.0	9	1.3	7.5

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
37	0.2381	206.0
38	0.2212	222.0
323	0.1479	314.0
342	0.1440	129.0
443	0.1417	200.0
36	0.1301	190.0
16	0.1227	288.0
24	0.1185	257.0
278	0.1172	277.0
216	0.1167	154.0

FIGURE 3 (CONT)

Spectrum # 33 Ret. Time= 5.0 Sample # 1 Total Abundance = 1937



Probably an isomer of di-t-butyl methylphenol

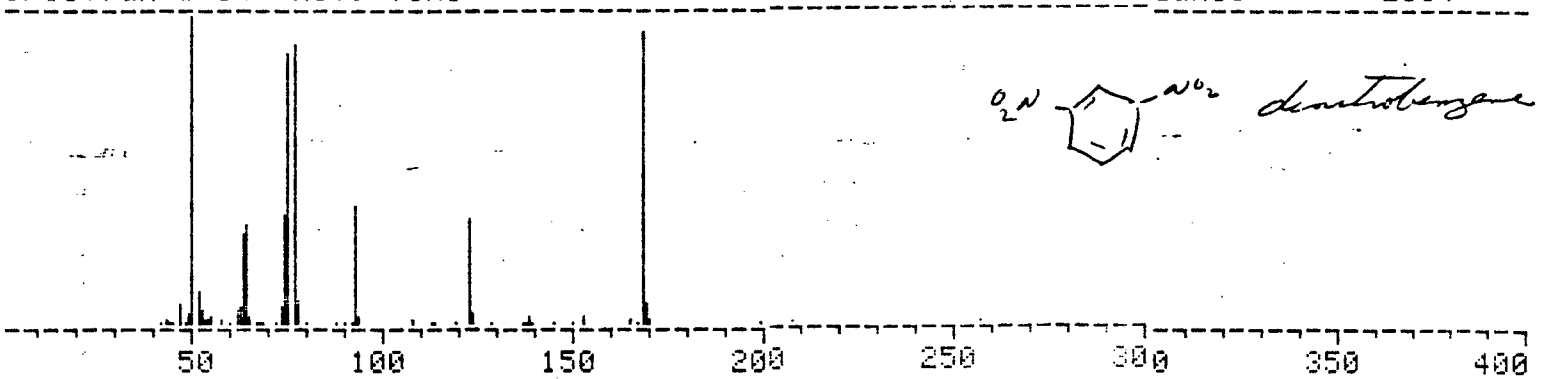
10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
41.0	167	100.0	35.5
67.0	67	40.1	23.3
135.0	55	32.9	38.5
149.0	47	28.1	36.3
161.0	58	34.7	48.4
163.0	51	30.5	43.1
177.0	109	65.3	100.0
203.0	22	13.2	23.1
205.0	27	16.2	28.7
220.0	75	44.9	85.5

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
484	0.6644	220.0
225	0.5797	152.0
236	0.5652	152.0
226	0.5509	152.0
298	0.5287	108.0
260	0.5241	154.0
254	0.5044	136.0
232	0.5032	154.0
211	0.4898	184.0
237	0.4830	152.0

Spectrum # 34 Ret. Time= 5.4 Sample # 1 Total Abundance = 2184



dinitrobenzene

FIGURE 3 (CONT)

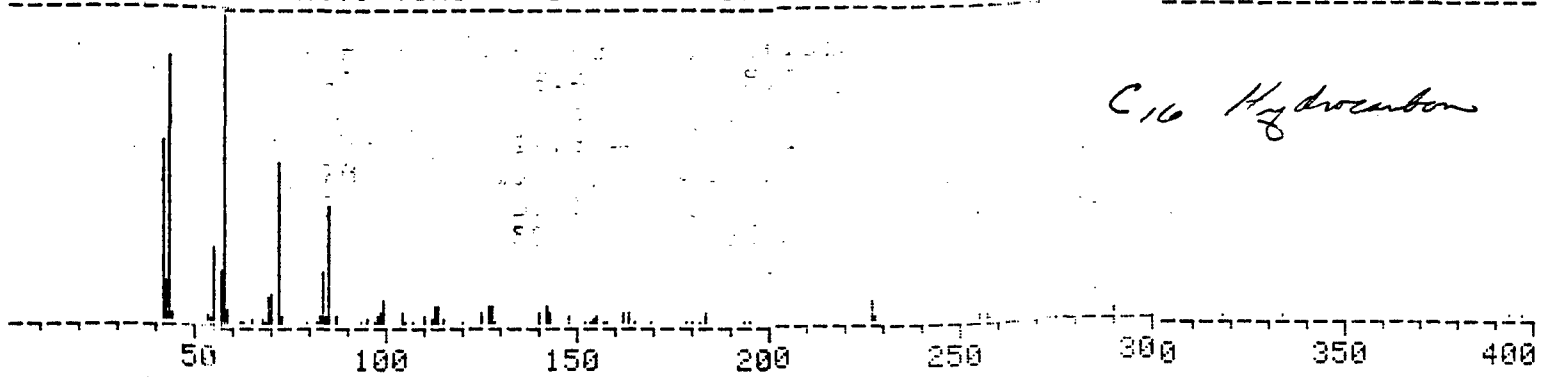
10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
50.0	333	100.0	31.8
63.0	95	28.5	11.4
64.0	107	32.1	13.1
74.0	115	34.5	16.2
75.0	291	87.4	41.6
76.0	299	89.8	43.4
92.0	126	37.8	22.1
122.0	112	33.6	26.1
168.0	312	93.7	100.0
169.0	23	6.9	7.4

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
368	0.4487	168.0
202	0.4039	260.0
61	0.3827	225.0
491	0.2827	103.0
277	0.2447	225.0
493	0.2301	180.0
20	0.2124	250.0
486	0.2087	124.0
450	0.2083	312.0
463	0.1962	438.0

Spectrum # 35 Ret. Time= 5.8 Sample # 1 Total Abundance = 624



C_{16} Hydrocarbon

10 peaks used for search:

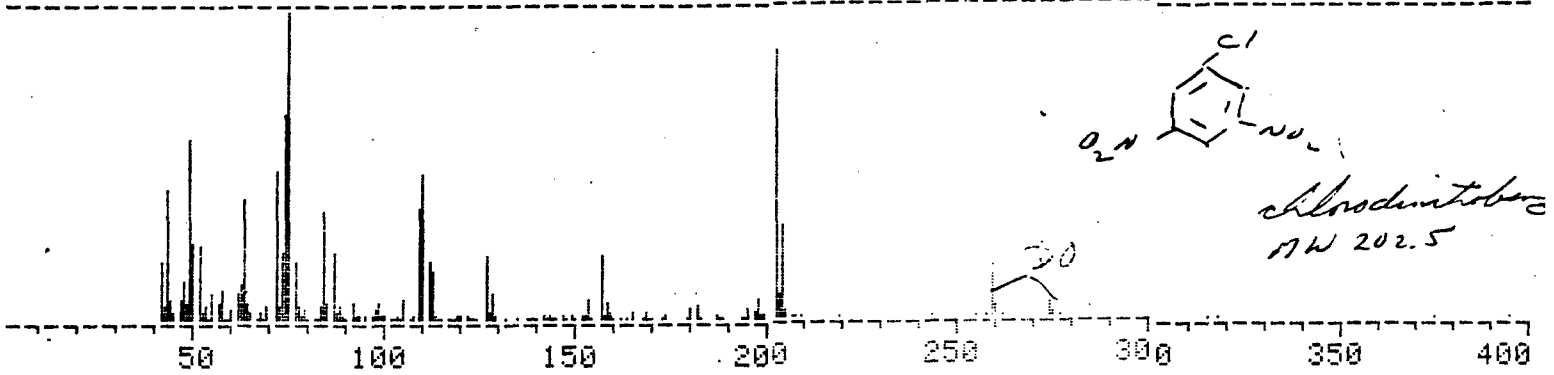
Mass	Linear Abund	% Abund	Significance
41.0	63	57.8	41.6
43.0	93	85.3	64.4
55.0	26	23.9	23.0
56.0	18	16.5	16.2
57.0	109	100.0	100.0
71.0	57	52.3	65.1
83.0	18	16.5	24.0
85.0	41	37.6	56.1
99.0	9	8.3	14.3
226.0	8	7.3	29.1

FIGURE 3 (cont'd)

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
5	0.9853	226.0
4	0.9853	212.0
3	0.9835	198.0
7	0.9782	254.0
6	0.9706	240.0
1	0.9687	170.0
8	0.9667	268.0
438	0.9620	312.0
437	0.9586	284.0
436	0.9546	256.0

Spectrum # 36 Ret. Time= 6.0 Sample # 1 Total Abundance = 4792



chlorodinitrobenzene
MW 202.5

10 peaks used for search:

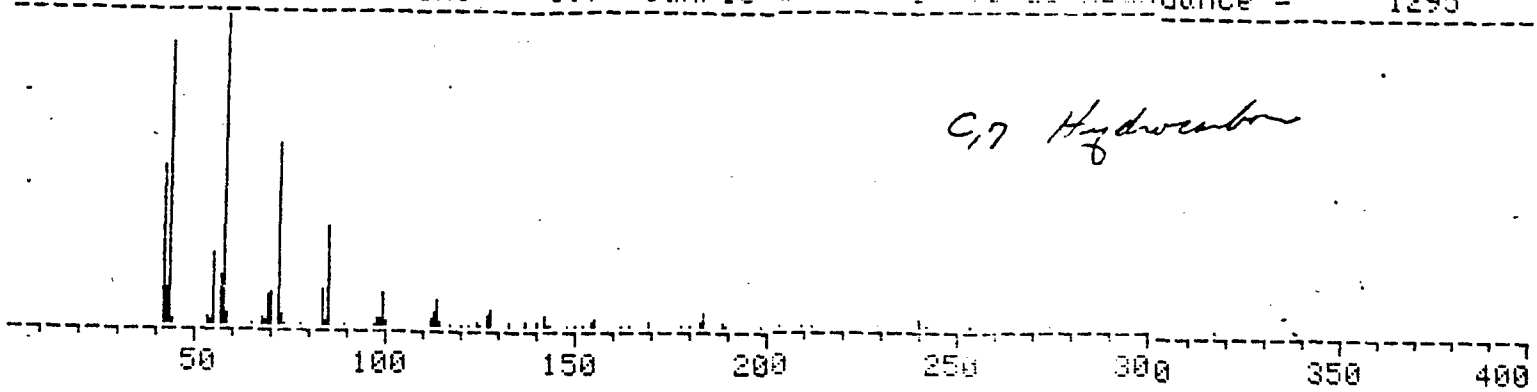
Mass	Linear Abund	% Abund	Significance
71.0	193	47.7	19.5
74.0	269	66.4	28.4
75.0	405	100.0	43.3
84.0	142	35.1	17.0
109.0	144	35.6	22.4
110.0	190	46.9	29.8
156.0	85	21.0	18.9
202.0	347	85.7	100.0
204.0	120	29.6	34.9
259.0	69	17.0	25.5

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
493	0.5389	180.0
202	0.5316	260.0
81	0.4548	202.0
330	0.4503	202.0
329	0.4463	202.0
450	0.3942	312.0
460	0.3763	466.0
286	0.3621	298.0
451	0.3616	326.0
448	0.3613	284.0

Figure 3 (con't)

Spectrum # 37 Ret. Time= 6.7 Sample # 1 Total Abundance = 1295



C_{17} Hydrocarbon

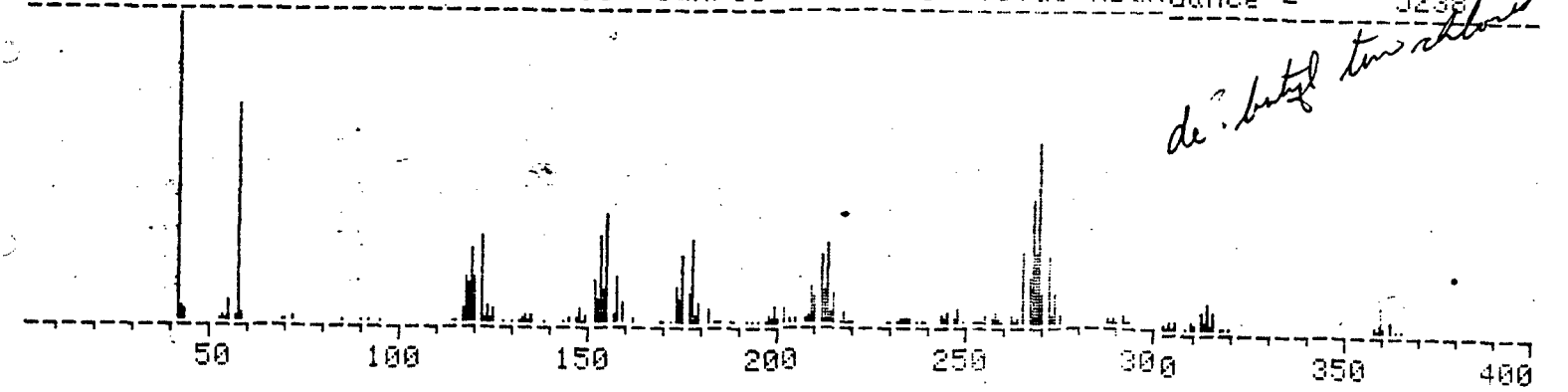
10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
41.0	120	51.3	36.9
43.0	210	89.7	67.7
55.0	54	23.1	22.3
56.0	37	15.8	15.5
57.0	234	100.0	100.0
71.0	136	58.1	72.4
83.0	27	11.5	16.8
85.0	74	31.6	47.2
99.0	25	10.7	18.6
113.0	21	9.0	17.8

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
5	0.9907	226.0
3	0.9897	198.0
4	0.9870	212.0
7	0.9866	254.0
6	0.9775	240.0
8	0.9766	268.0
1	0.9758	170.0
438	0.9634	312.0
437	0.9571	284.0
436	0.9541	256.0

Spectrum # 38 Ret. Time= 7.1 Sample # 1 Total Abundance = 5238



di-butyl terephthalate

FIGURE 3 (CON'T)

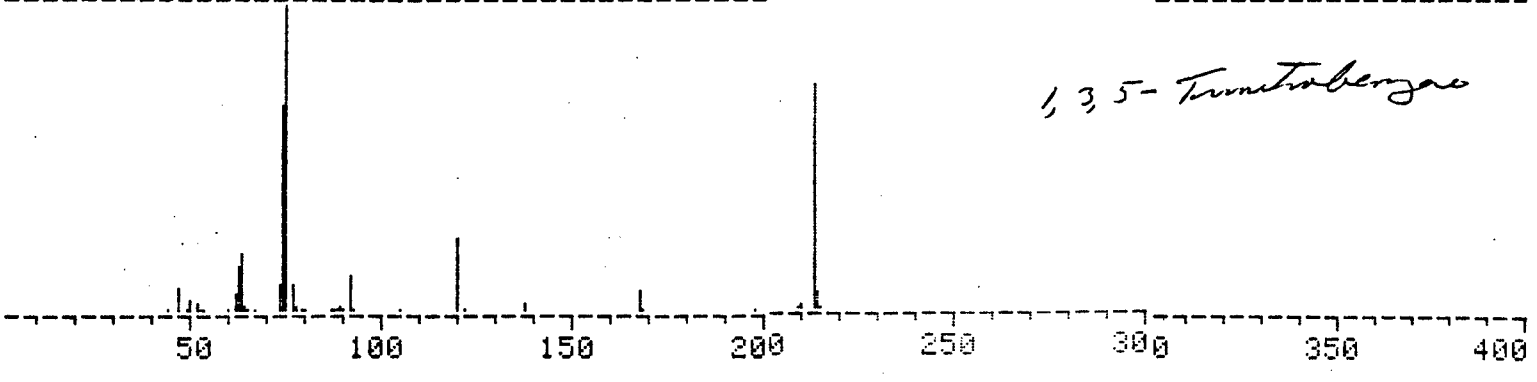
10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
153.0	141	47.6	27.1
155.0	179	60.5	34.8
177.0	135	45.6	30.0
211.0	113	38.2	29.9
213.0	130	43.9	34.8
265.0	115	38.9	38.3
267.0	203	68.6	68.1
268.0	115	38.9	38.7
269.0	296	100.0	100.0
271.0	113	38.2	38.5

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
372	0.2332	186.0
280	0.2107	278.0
349	0.1943	154.0
484	0.1919	220.0
469	0.1899	153.0
277	0.1565	225.0
513	0.1533	266.0
403	0.1460	296.0
134	0.1447	192.0
109	0.1419	320.0

Spectrum # 39 Ret. Time= 7.8 Sample # 1 Total Abundance = 10207



10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
62.0	378	14.8	5.9
63.0	487	19.1	7.8
74.0	1695	66.6	31.8
75.0	2546	100.0	48.4
76.0	221	8.7	4.3
91.0	311	12.2	7.2
120.0	606	23.8	18.4
167.0	188	7.4	8.0
213.0	1852	72.7	100.0
214.0	145	5.7	7.9

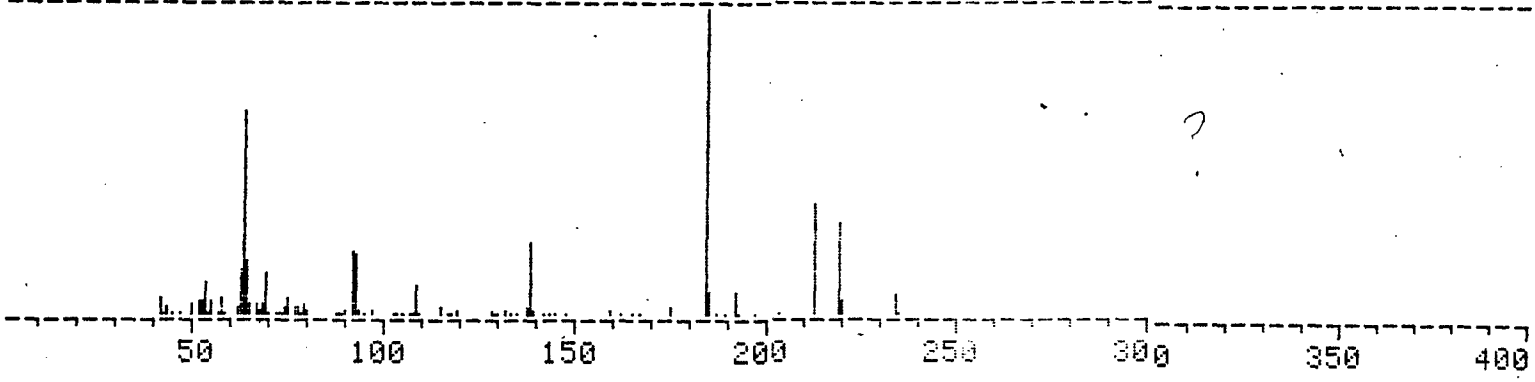
10 BEST MATCHES: Library #2

Figure 3 (cont'd)

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
202	0.6360	260.0
493	0.4772	180.0
450	0.4726	312.0
447	0.4487	256.0
286	0.4343	298.0
451	0.4335	326.0
448	0.4333	284.0
449	0.4272	298.0
463	0.4245	438.0
284	0.4236	242.0

Spectrum # 40 Ret. Time= 8.1 Sample # 1 Total Abundance = 7031



10 peaks used for search:

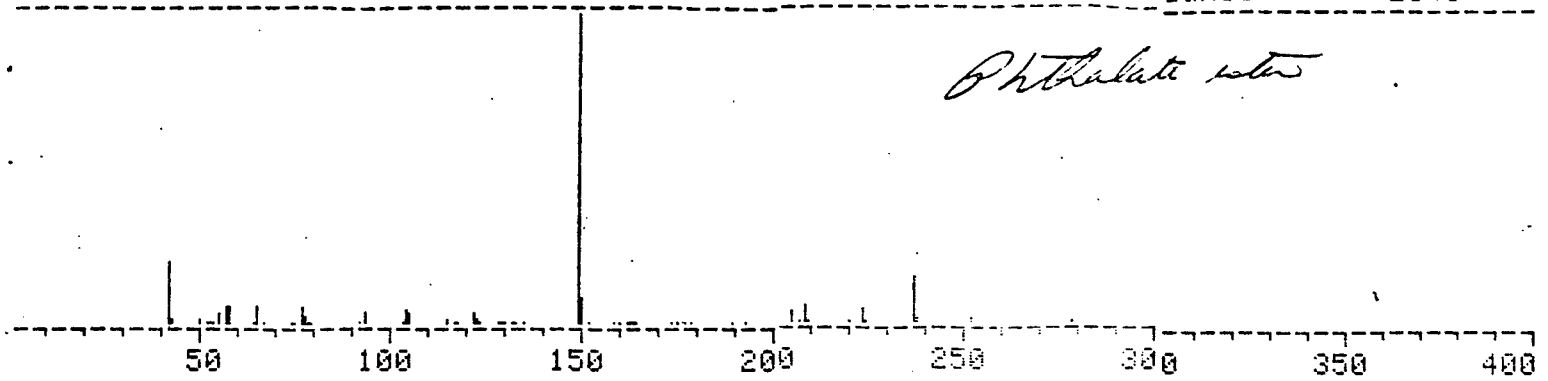
Mass	Linear Abund	% Abund	Significance
63.0	871	66.3	22.7
91.0	273	20.8	10.3
92.0	265	20.2	10.1
138.0	310	23.6	17.7
184.0	1313	100.0	100.0
185.0	103	7.8	7.9
191.0	109	8.3	8.6
212.0	475	36.2	41.7
219.0	397	30.2	36.0
234.0	96	7.3	9.3

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
468	0.3907	186.0
501	0.2690	162.0
481	0.2545	172.0
429	0.2447	182.0
486	0.2378	124.0
519	0.2307	0.0
41	0.2284	195.0
466	0.2259	142.0
369	0.2184	186.0
86	0.2122	230.0

FIGURE 3 (CONT)

Spectrum # 41 Ret. Time= 10.3 Sample # 1 Total Abundance = 2040



10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
41.0	153	20.1	5.5
104.0	41	5.4	3.8
149.0	760	100.0	100.0
150.0	72	9.5	9.5
205.0	30	3.9	5.4
208.0	42	5.5	7.7
223.0	40	5.3	7.9
236.0	113	14.9	23.5
237.0	17	2.2	3.6
251.0	18	2.4	4.0

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
296	0.9085	222.0
297	0.8323	390.0
304	0.7272	220.0
198	0.3220	275.0
319	0.2997	316.0
484	0.2680	220.0
426	0.1953	220.0
187	0.1741	275.0
205	0.1658	299.0
225	0.1584	152.0

Spectrum # 42 Ret. Time= 10.6 Sample # 1 Total Abundance = 1729

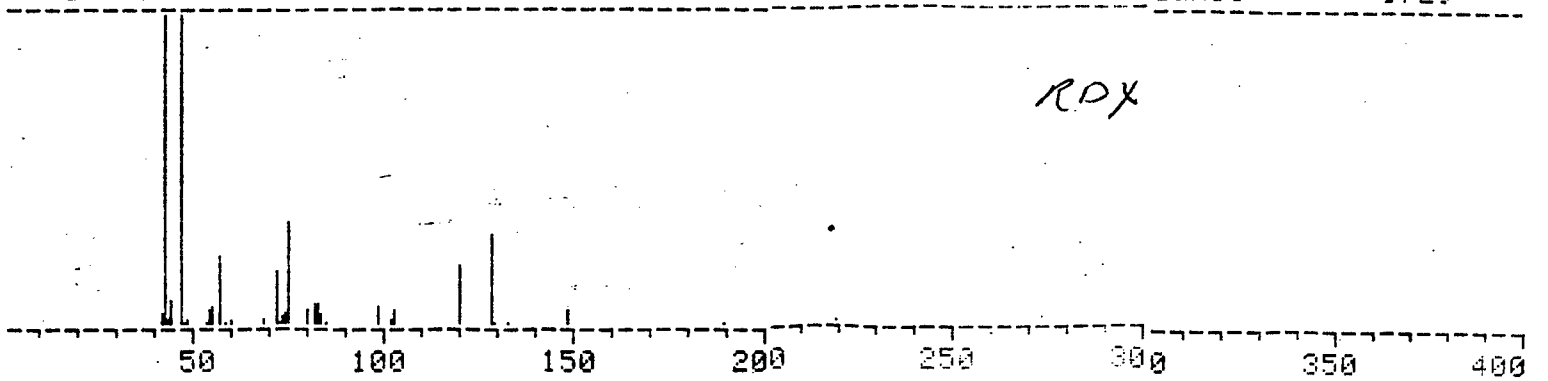


FIGURE 3 (cont'd

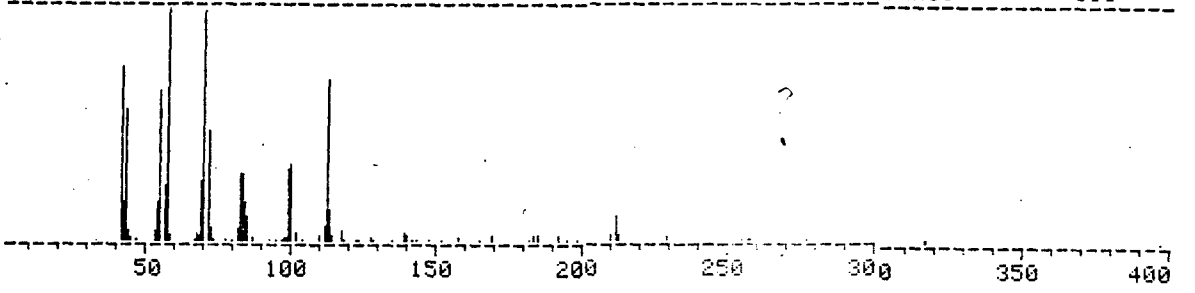
10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
42.0	409	98.6	90.0
46.0	415	100.0	100.0
56.0	93	22.4	27.3
71.0	72	17.3	26.8
75.0	138	33.3	54.2
82.0	27	6.5	11.6
98.0	25	6.0	12.3
120.0	78	18.8	49.0
128.0	122	29.4	81.8
148.0	22	5.3	17.1

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
133	0.2749	164.0
467	0.2596	138.0
202	0.2035	260.0
348	0.1834	128.0
135	0.1823	145.0
341	0.1164	101.0
46	0.1152	248.0
106	0.1095	177.0
196	0.1025	229.0
483	0.0895	122.0

Spectrum # 43 Ret. Time= 17.1 Sample # 1 Total Abundance = 851



10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
41.0	70	73.7	38.8
55.0	61	64.2	45.4
57.0	95	100.0	73.3
70.0	93	97.9	88.1
71.0	45	47.4	43.2
82.0	28	29.5	31.1
83.0	28	29.5	31.4
99.0	29	30.5	38.8
100.0	31	32.6	41.9
112.0	66	69.5	100.0

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight
247	0.7103	366.0
287	0.7050	130.0
246	0.6212	332.0
8	0.6206	268.0
6	0.6202	240.0
193	0.6088	380.0
437	0.6059	284.0
436	0.6020	256.0
438	0.5969	312.0
2	0.5943	184.0

FIGURE 4. Methyl derivatization of LAAP Lagoon Water

```
PEAKFINDER PROGRAM  [Rev 10/9/78]

>> CURRENT GC CONDITIONS:  Oven=106.0 Inj. Port = 44.0 Retention time=   0.0


Last spectrum recorded was  18  Total Available = 100

Next Spectrum recorded will be   1

** CONDITIONS FOR RUN #     1  dated: 5/31/1980  Saturday
*************************************************************************
 TEMP1   TIME1     RATE     TEMP2  TIME2  INJ.PORT  MAX.OVEN  SOLVENT  RUN TIME
  Deg.    min.  Deg/min.    Deg.    Min.     Deg.      Deg.      Min.      Min.
  140     0.0     15.0       260    32.0     210       280       0.0      30.0

MS PEAK DETECT THRESHOLD =     5.0 linear counts
FLOW RATE =                   16   ml/min
SAMPLES PER .1 AMU =           8   SCAN SPEED = 200 amu/sec
ELECTRON MULTIPLIER =       1800   volts
GC PEAK DETECT THRESHOLD =   400   TRIGGERED ON TOTAL ABUNDANCE
REAL TIME STRIPPING OF VALLEYS FROM PEAKS
```

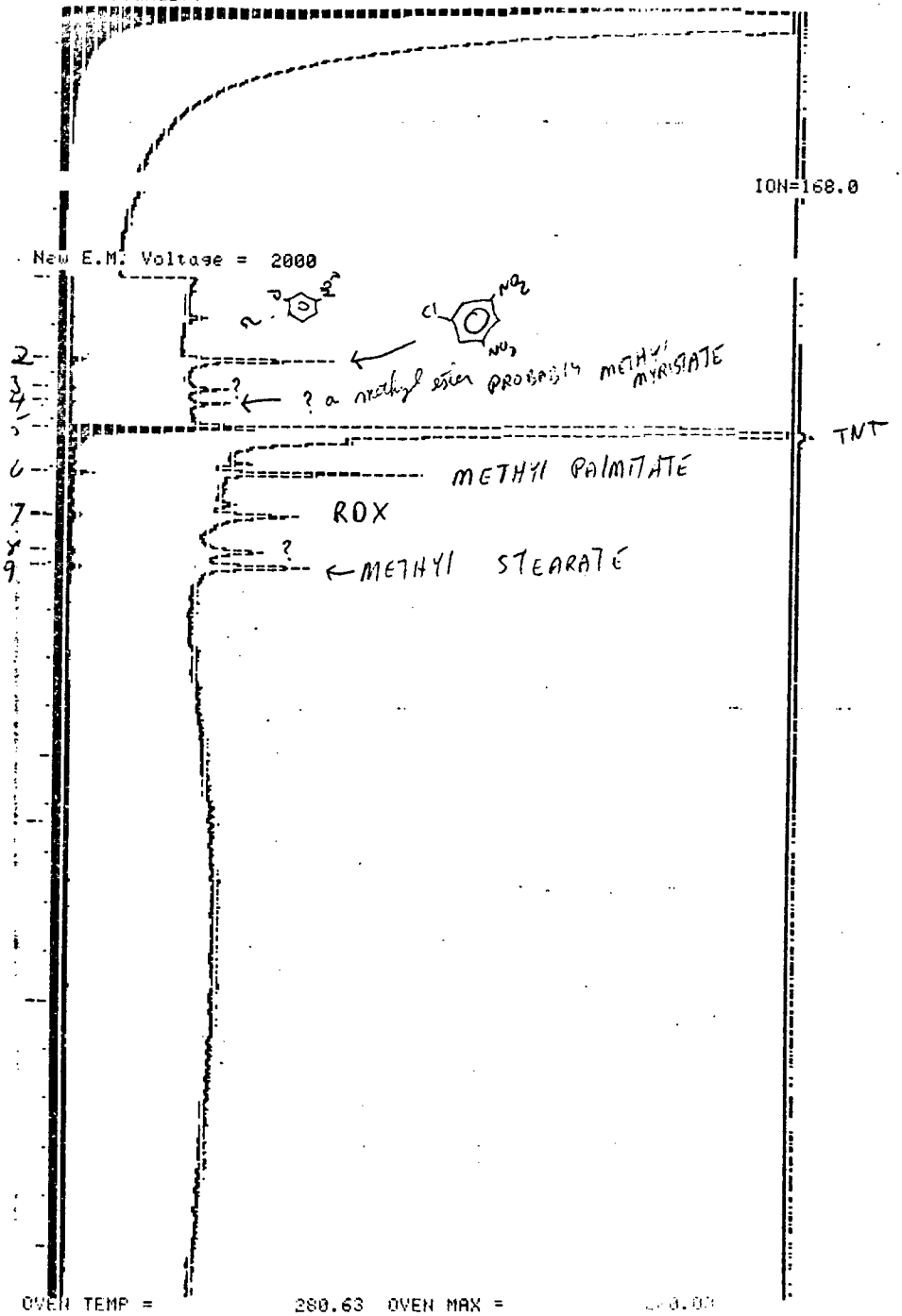
FIGURE 4 (cont'd)

SAMPLE NAME LAAP Lagoon - Acid Extract - BE-M014

OPERATOR BT

TOTAL ABUNDANCE FROM 40 TO 460 amu
Full Scale= 9920
Excess Source Pressure!
MS in Standby!
No Emission Current!
Detector Problem!
MS in Standby!

ION 121.0
Full Scale= 150



OVEN TEMP = 280.63 OVEN MAX = 0.00
RUN TERMINATED (OVEN COOLED)

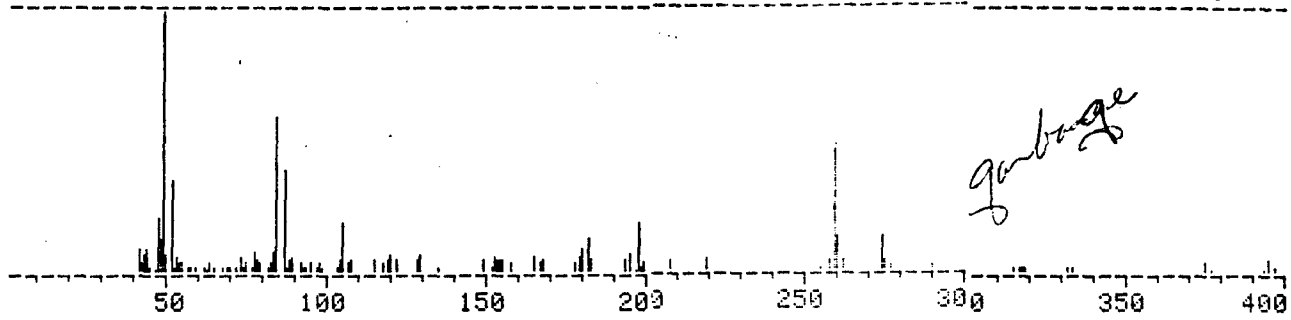
FIGURE 4 (CONT)

SPECTRA SAVED: Run # 1

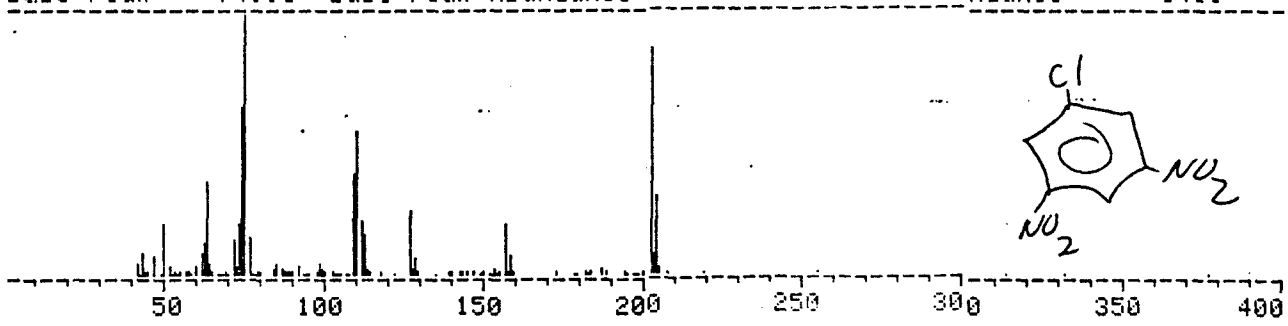
Spectrum	Ret. Time	Total Abund.	Relative Abund.	Base Peak
1	4.9	635	3.9%	48.90
2	6.6	1431	8.7%	74.95
3	7.3	389	2.4%	235.10
4	7.5	369	2.2%	73.95
5	8.1	16409	100.0%	209.95
6	9.0	1657	10.1%	73.95
7	9.9	648	3.9%	41.90
8	10.7	555	3.4%	40.90
9	11.0	849	5.2%	73.95
10	16.3	428	2.6%	72.95

Spectra Plot/Tab Program: [Rev 8/4/78]

** Spectrum # 1 ** Sample # 1 Retention Time = 4.9 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 105
File type = linear
Base Peak = 48.90 Base Peak Abundance = 85 Total Abundance = 635



** Spectrum # 2 ** Sample # 1 Retention Time = 6.6 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 112
File type = linear
Base Peak = 74.95 Base Peak Abundance = 183 Total Abundance = 1431



** Spectrum # 3 ** Sample # 1 Retention Time = 7.3 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 108
File type = linear
Base Peak = 235.10 Base Peak Abundance = 36 Total Abundance = 389

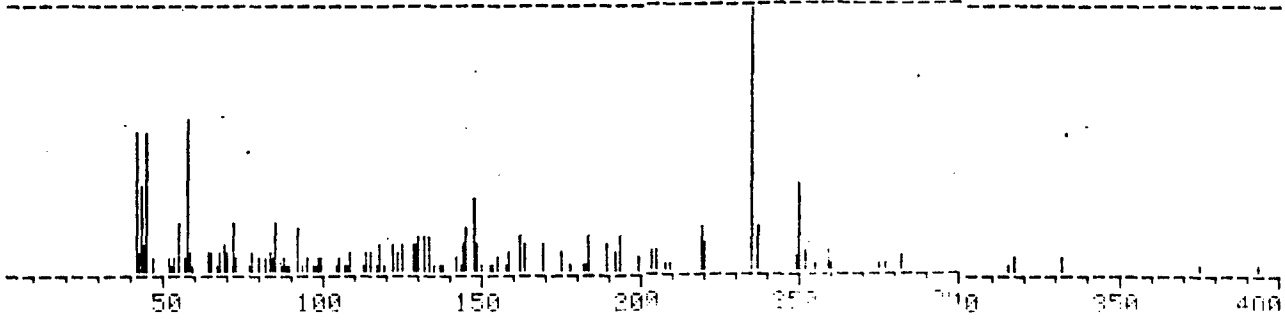
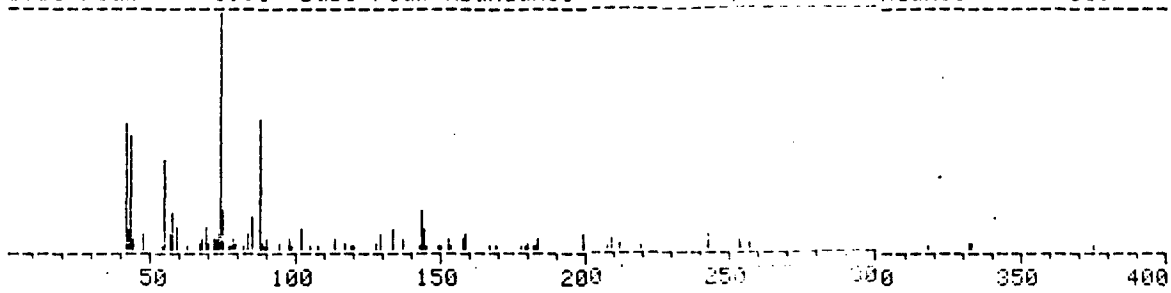
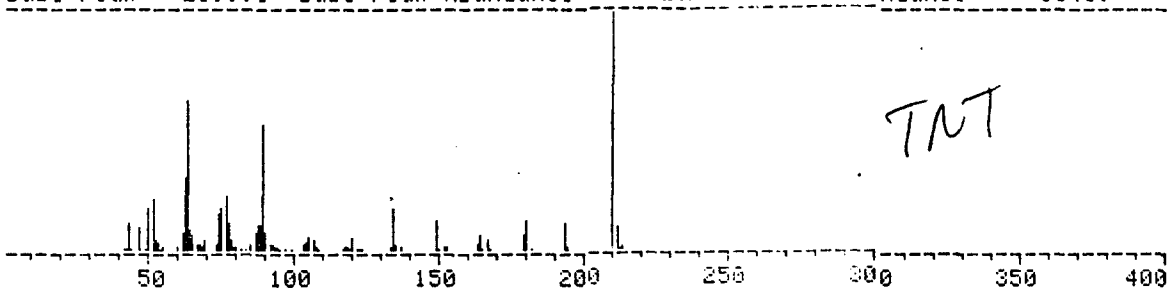


Figure 4 (cont'd)

** Spectrum # 4 ** Sample # 1 Retention Time = [illegible] minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 97
File type = linear
Base Peak = 73.95 Base Peak Abundance = 58 Total Abundance = 369

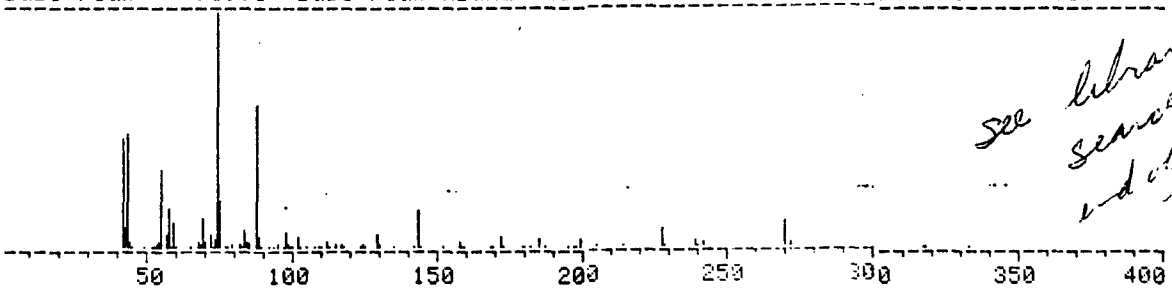


** Spectrum # 5 ** Sample # 1 Retention Time = 8.1 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 144
File type = linear
Base Peak = 209.95 Base Peak Abundance = 2520 Total Abundance = 16409



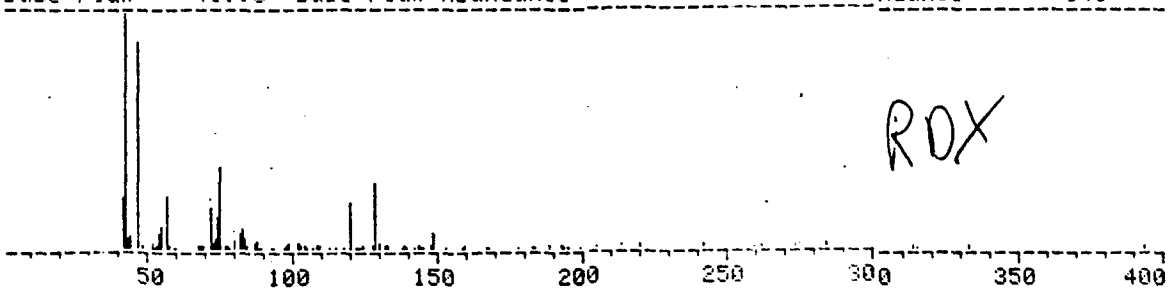
TNT

** Spectrum # 6 ** Sample # 1 Retention Time = 9.0 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 121
File type = linear
Base Peak = 73.95 Base Peak Abundance = 295 Total Abundance = 1657



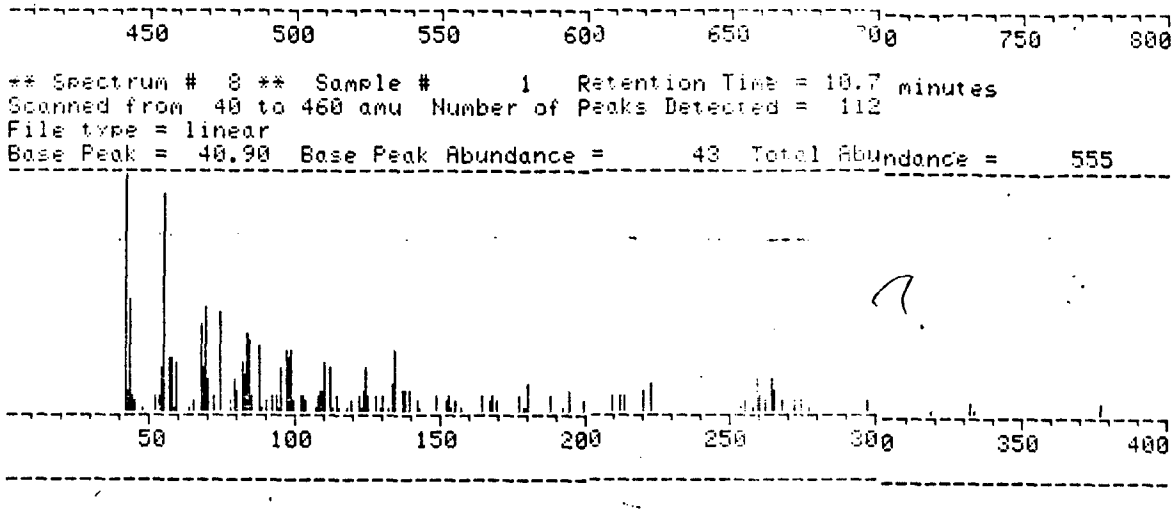
see library search at end of figure

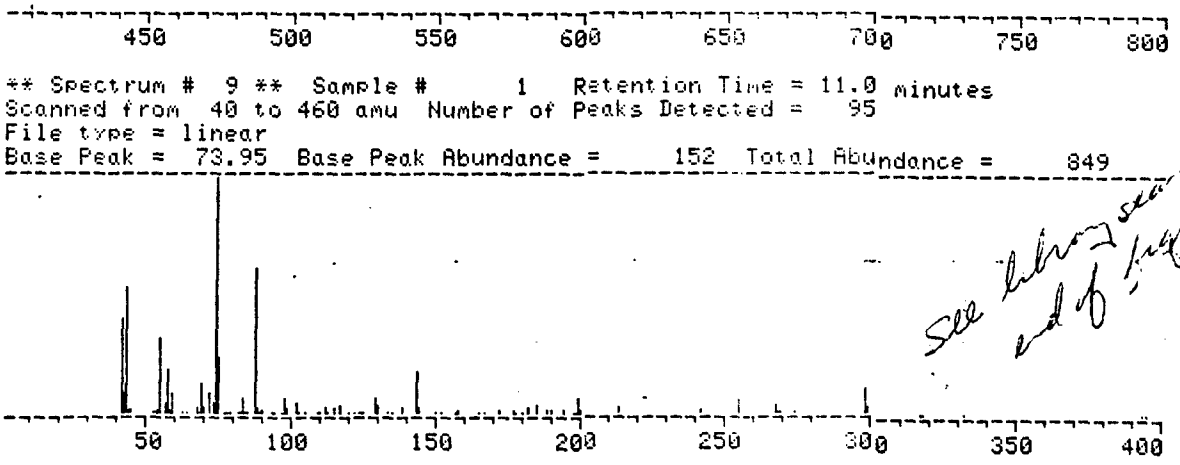
** Spectrum # 7 ** Sample # 1 Retention Time = 9.9 minutes
Scanned from 40 to 460 amu Number of Peaks Detected = 97
File type = linear
Base Peak = 41.90 Base Peak Abundance = 122 Total Abundance = 648



RDX

FIGURE 4 (CONT)





see library search at end of figure

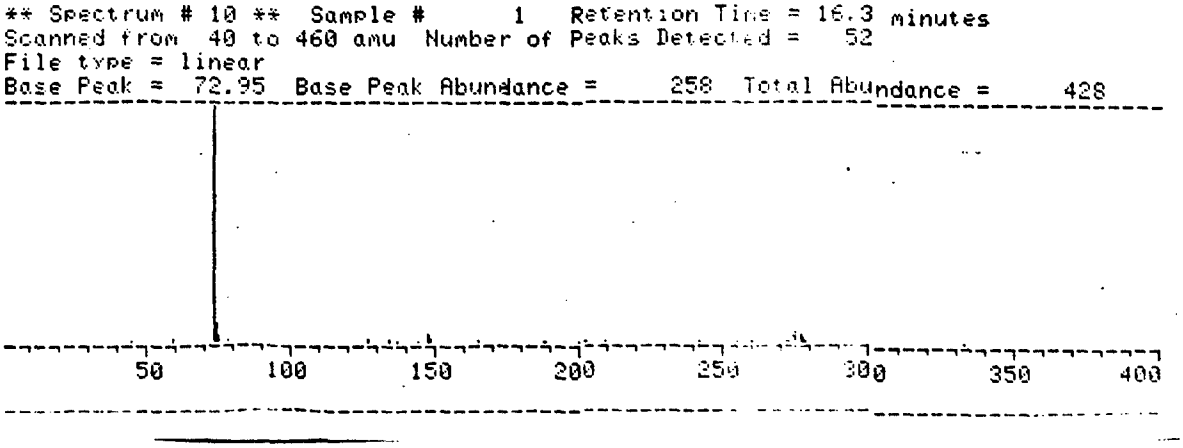
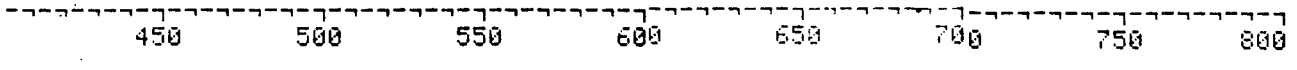
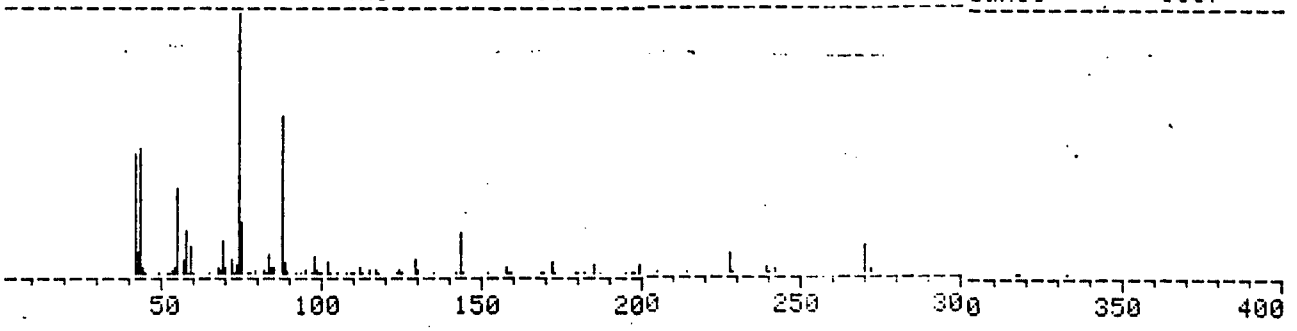


FIGURE 4 (CONT.)



*** LIBRARY SEARCH [rev. 1/1/78]

Spectrum # 6 Ret. Time= 9.0 Sample # 1 Total Abundance = 1657



10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
41.0	135	45.8	25.4
43.0	142	48.1	28.0
55.0	96	32.5	24.2
57.0	51	17.3	13.3
74.0	295	100.0	100.0
75.0	58	19.7	19.9
87.0	177	60.0	70.5
143.0	48	16.3	31.4
227.0	23	7.8	23.9
270.0	33	11.2	40.8

10 BEST MATCHES: Library #2

Entry	Similiarity Index	Molecular Weight	
446	0.9913	228.0	
285	0.9836	270.0	← methyl palmitate
284	0.9830	242.0	
448	0.9823	284.0	
286	0.9815	298.0	
457	0.9799	354.0	
447	0.9792	256.0	
283	0.9782	214.0	
454	0.9768	340.0	
458	0.9761	368.0	

Spectrum # 9 Ret. Time= 11.0 Sample # 1 Total Abundance = 849

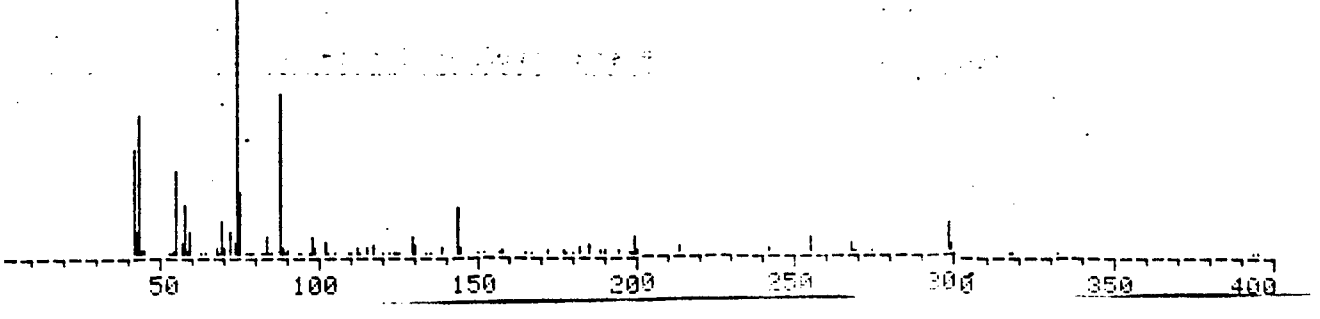


FIGURE 4 (cont.)

10 peaks used for search:

Mass	Linear Abund	% Abund	Significance
41.0	61	40.1	22.2
43.0	81	53.3	31.0
55.0	49	32.2	24.0
74.0	152	100.0	100.0
75.0	36	23.7	24.0
87.0	92	60.5	71.2
143.0	27	17.8	34.3
199.0	10	6.6	17.7
255.0	9	5.9	20.4
298.0	16	10.5	42.4

10 BEST MATCHES: Library #2

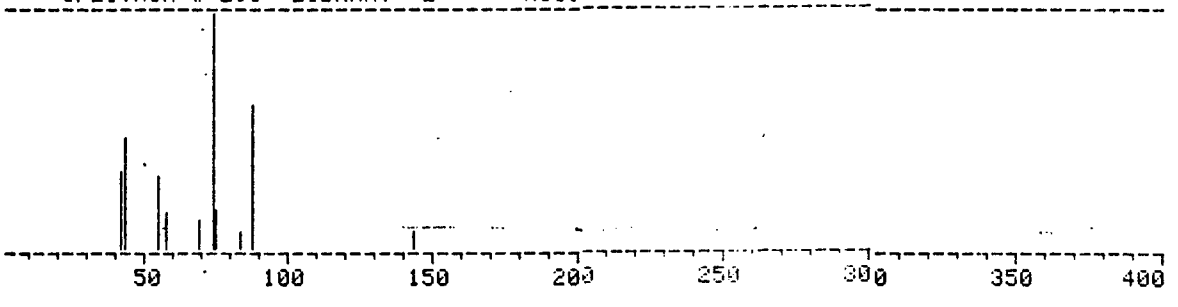
Entry	Similiarity Index	Molecular Weight
446	0.9837	228.0
285	0.9772	270.0
448	0.9770	284.0
447	0.9755	256.0
284	0.9740	242.0
286	0.9731	298.0 — methyl stearate
449	0.9725	298.0 — methyl isostearate
457	0.9716	354.0
281	0.9694	186.0
283	0.9691	214.0

HP 5992 SYSTEM OPTIONS: [Rev.11/20/78]

1 = AUTOTUNE
2 = PEAKFINDER
3 = EDIT MASS SPEC OPERATING PARAMETERS
4 = LIBRARY SEARCH
5 = PLOT/TABULATE
6 = PRINT TAPE LAYOUT
7 = PLOT SPECTRA ON X-Y PLOTTER
8 = SHOW AND EDIT LIBRARIES
9 = RESIZE SPECTRAL FILES
10 = DFTPP NORMALIZER
11 = SPECTRUM MANIPULATION PROGRAM

Library Editing Program [rev 7/26/78]

*** SPECTRUM # 285 LIBRARY 2 *** Mol. Weight= 270.0



*** SPECTRUM # 286 LIBRARY 2 *** Mol. Weight= 298.0

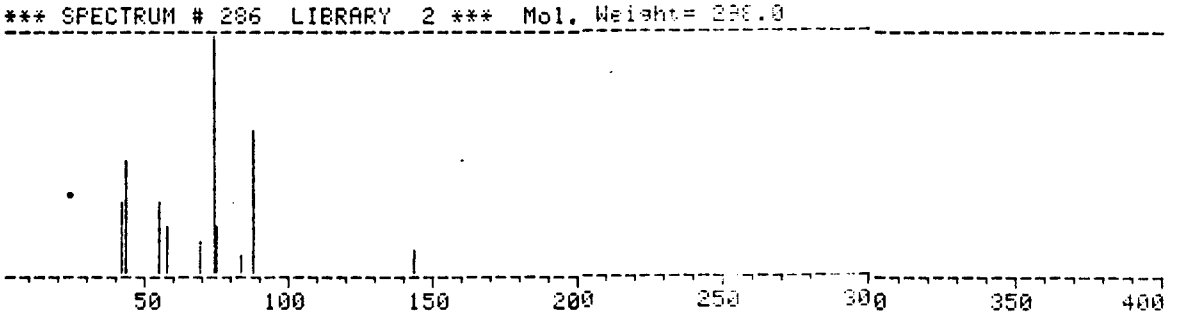


Figure 5 TNT · SAKM

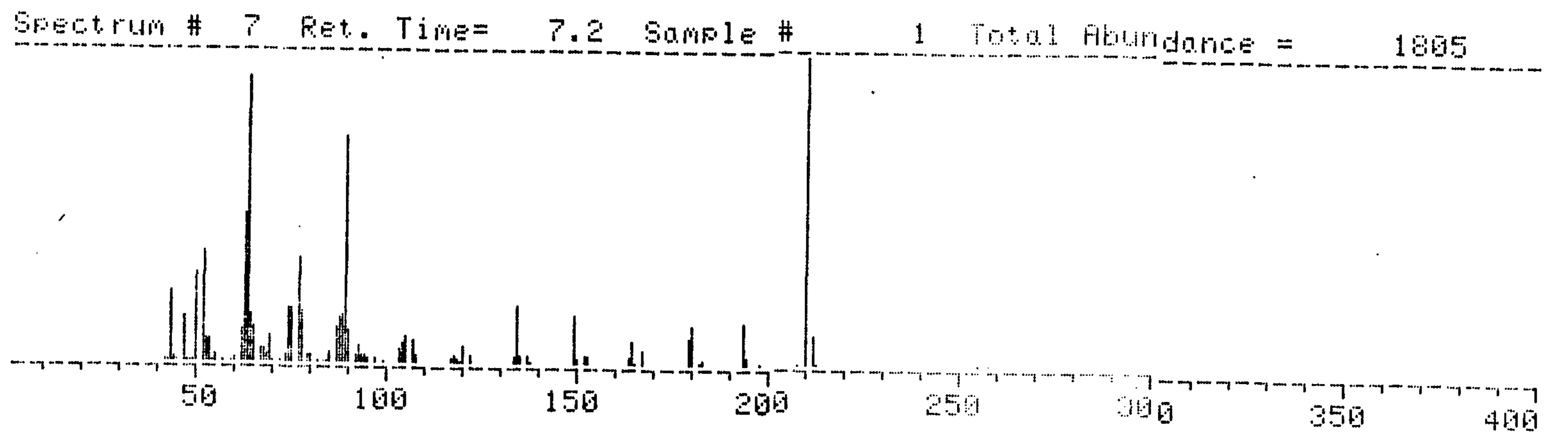
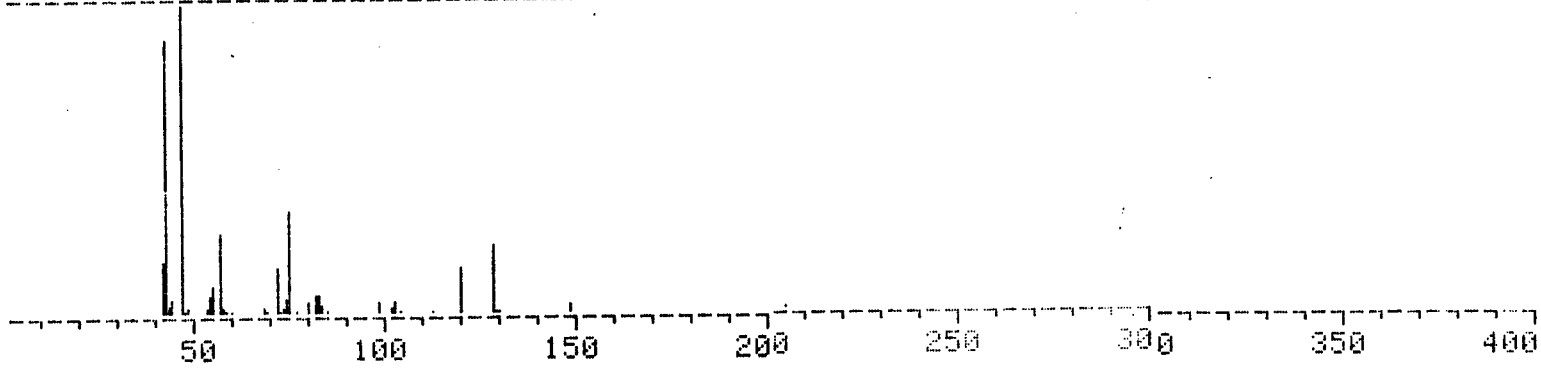


Figure 6. Mass Spectrum

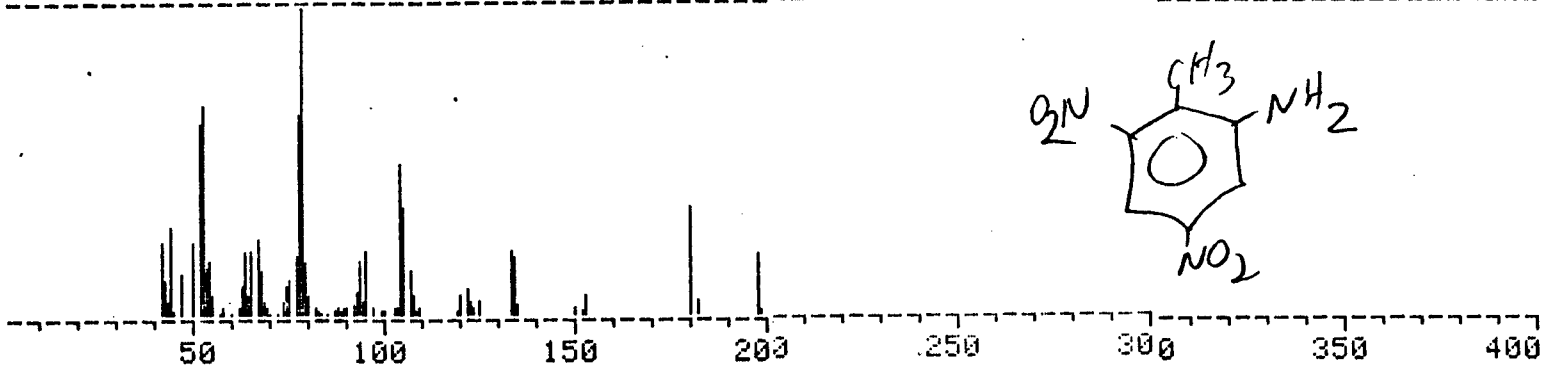
*** LIBRARY SEARCH [rev. 1/1/78]

Spectrum # 4 Ret. Time= 9.7 Sample # 1 Total Abundance = 5227



Fraction 4 2-amino-dinitrotoluene

Spectrum # 32 Ret. Time= 11.8 Sample # 1 Total Abundance = 1458



Spectrum # 33 Ret. Time= 8.6 Sample # 1 Total Abundance = 4585

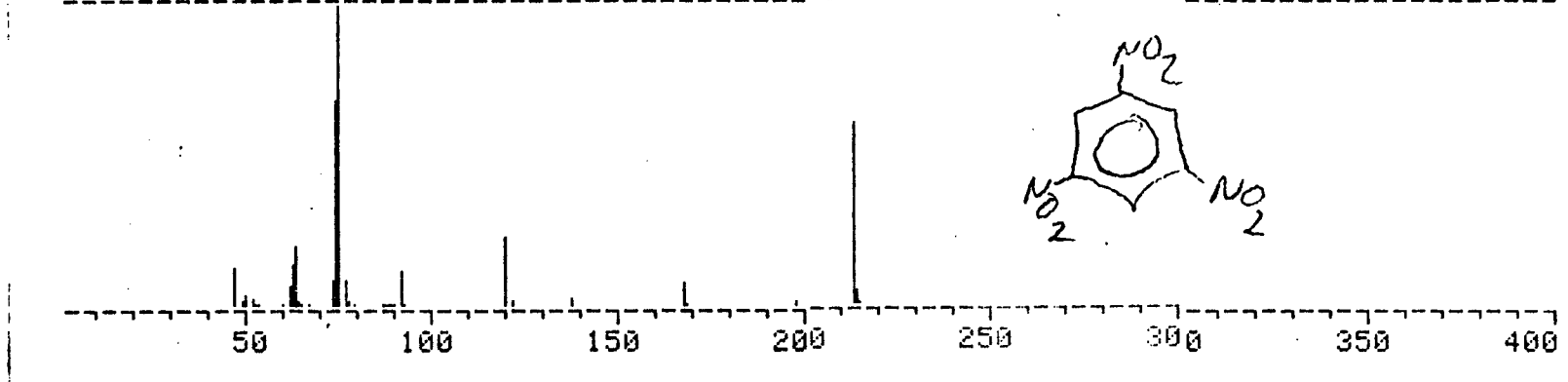
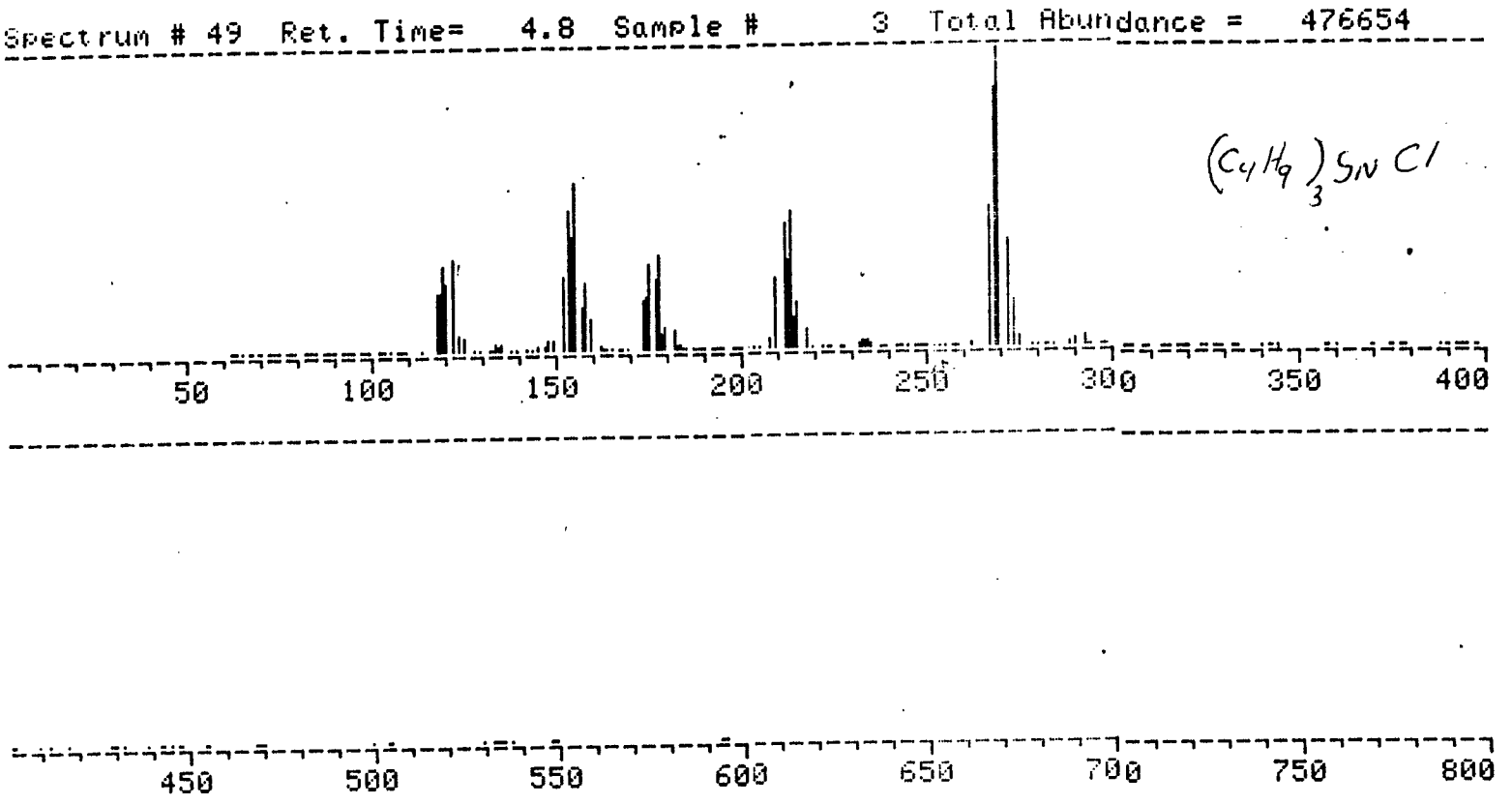
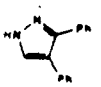
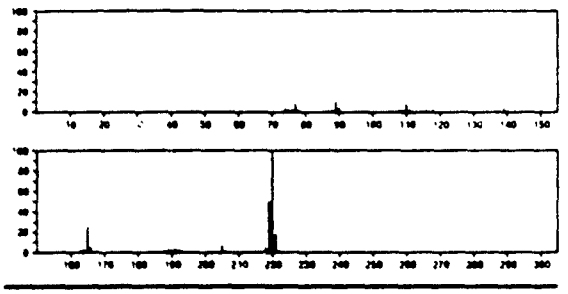


Figure 9 Tributyltin Chloride

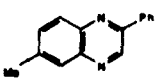


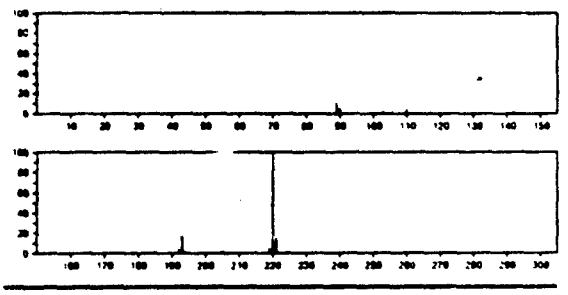
220 $C_{15}H_{12}N_2$ 24567-08-6
1*H* Pyrazole, 3,4-diphenyl



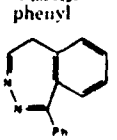


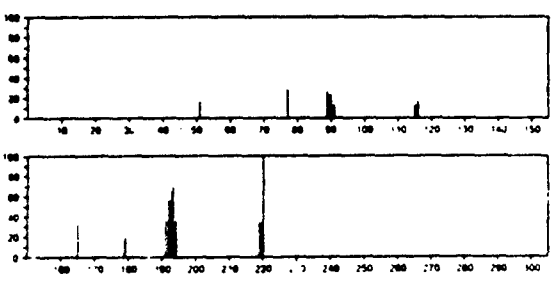
220 $C_{15}H_{12}N_2$ 25187-18-2
Quinoxaline, 6-methyl-2-phenyl



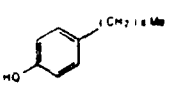


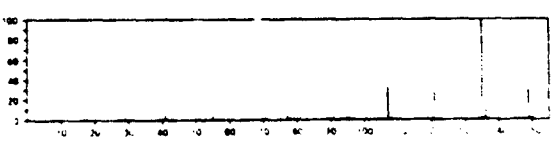
220 $C_{15}H_{12}N_2$ 52095-33-7
5*H*-2,3-Benzodiazepine, 1-phenyl

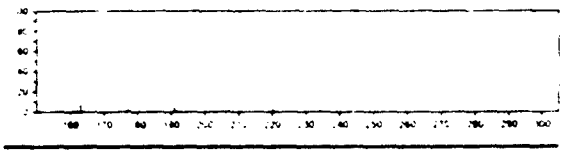




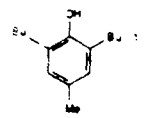
220 $C_{15}H_{24}O$ 104-40-5
Phenol, 4-nonyl

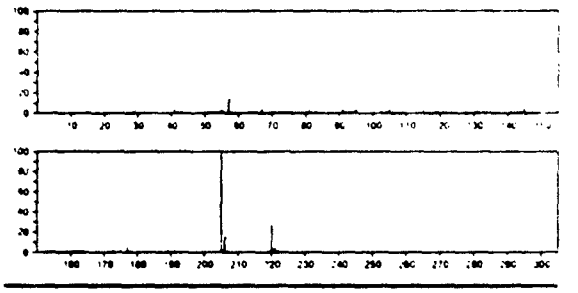




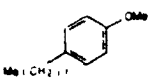


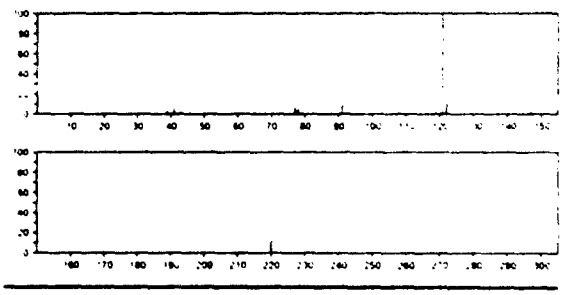
220 $C_{15}H_{24}O$ 128-[illegible]
Phenol, 2,6-bis(1,1-dimethylethyl)-4-methyl-





220 $C_{15}H_{24}O$ 5307-19-5
Anisole, *p*-octyl-





220 $C_{15}H_{24}O$ 1300[illegible]-45-1
Santalol

STRUCTURE UNDEFINED